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Investigation of Reactivity of
Titanium and other Materials with
Liquid Oxygen and Nitrogen Tetroxide

Final Report

Contract No. NAS8-20078

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INCORPORATED
RESEARCH AND ENGINEERING DEPARTMENT
MURRAY HILL, NEW JERSEY

RE-66-111-CRE-35

Investigation of Reactivity of Titanium and other Materials with Liquid Oxygen and Nitrogen Tetroxide

Final Report

to

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Huntsville, Alabama 35812
Attention: PR-EC

Contract No. NAS8-20078
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ABSTRACT

This report summarizes the development and application of equipment and methods for determination of the energy required to initiate reaction of oxygen or nitrogen tetroxide with various engineering materials by one mode of energy input, namely adiabatic compression.

The year's work was divided into three main categories:

1) A survey of background literature;

2) Design, fabrication and evaluation of a piston type adiabatic compressor apparatus with the capabilities of operating in a temperature range from -183.0°C (-297.4°F) to 150°C (302°F) and attaining a maximum pressure of 30,000 p.s.i. within the sample chamber; and

3) An experimental test and data evaluation program which was limited due to the emphasis placed on attaining the required testing parameters and damaging effects to the tester when reactions occurred between an oxidizer and the material under test. A recommendation is presented in this report which would limit the damaging effects of a reaction to expendable parts.

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1. INTRODUCTION

In specifying propellant systems for launch vehicles many factors are considered before the propellants are determined to be operational. Among these factors is the determination of the compatibility of the propellant with the materials of construction when subjected to external stimuli. Liquid oxygen and nitrogen tetroxide currently are operational and, in all probability will continue to be used for future space missions; at present they are utilized in the main and auxiliary propulsion systems of Saturn.

In spite of the knowledge gained over many years in characterizing these particular oxidizers, there still are areas of information which are void and which prevent their usage with absolute confidence. Although considerable knowledge has been experienced in handling these particular oxidizers, the problem is one of chemical combination with various engineering materials which come in contact with the oxidizers that could and do result in a rapid release of energy under certain conditions.

Under this contract a useful tool, an improved adiabatic compression test apparatus, has been developed to study and determine the energy required to initiate a reaction when various engineering materials come in contact with these oxidizers, thus simulating conditions which might occur in an actual propellent system. With the knowledge obtained from the use of this device and correlated findings attained from other propellant evaluation methods, estimates as to the likelihood of probability of reaction can be more closely predicted.

Included in this report is a description of the design of the apparatus, solutions to problems encountered in utilizing the equipment over a wide temperature range, and a report of the results obtained. In addition there is included a statistical approach to the conduct of experiments for materials subjected to test; a literature survey conducted early in the program is presented in an Appendix.

2. PROGRAM REQUIREMENTS

The requirements of this project were two-fold; the development of the apparatus and the use of that apparatus in the evaluation of specified materials. Each of these requirements as set forth in the contract scope of work and subsequently modified are presented below.

2.1 APPARATUS REQUIREMENTS

A piston type adiabatic compression tester, as distinct from the U-tube type, was to be designed and developed with suitable instrumentation that would permit the determination of sensitivity of various engineering materials with three oxidizers. Specific requirements follow.

2.1.1 Oxidizers

The oxidizers which the tester was to be designed to handle include:

1) Gaseous air
2) Gaseous and liquid oxygen
3) Vapor and liquid nitrogen tetroxide

2.1.2 Engineering Materials

The engineering materials which were to be capable of being handled include:

1) Titanium alloy 5 Al - 2.5 Sn and 6 Al - 4 V
2) Aluminum alloy 5052-H-32
3) Magnesium HK 31 X A
4) Narmco "C" adhesive
5) Dow Corning oil FS 1281
6) n-Hexane
7) Dow Corning DC-33 grease
8) Bestoil

2.1.3 Apparatus Characteristics

The apparatus was to have the following capabilities:

1) Operate from liquid oxygen temperature, -183°C (-197.4°F) to 150°C (302°F).

2) Be constructed of a stainless steel alloy not subject to corrosion in contact with nitrogen tetroxide.

3) Be of a double wall construction to provide for cooling or heating of the apparatus.

4) The maximum driving pressure should be sufficient to give a maximum static pressure in the sample chamber of approx. 30,000 p.s.i. However, sufficient pressure should be available to insure 50% probability of reaction of 0.010 in. thick samples of a titanium alloy in liquid oxygen. Also the device must be capable of initiating reactions of Dow Corning Oil FS 1281 (10,000 CS) and liquid oxygen.

5) Methods incorporated in its design to vary the rate of pressurization.

6) Suitable burst diaphragms or other devices should be included in the design to preclude damage to the apparatus when an explosion occurs.

7) The total sample chamber volume should be approx. 5 ml.

8) The sample chamber should accommodate both liquid and solid samples from 1 mil to 0.250 in. thick.

9) The specimen cup holding device should be of an expendable nature and easily removable.

10) A bubble volume of 0.1 - 3 ml. should be accommodated.

11) A method by which a locked stroke motion can be achieved in order to prevent or limit piston bounce.

2.2 EXPERIMENTAL REQUIREMENTS

Equipment calibration tests were to be performed to determine the operational characteristic of the tester. Among the characteristics to be determined were the following:

1) Size of orifices for controlling rate of pressure rise,

2) Operational characters for the temperature control system,

3) Position of piston for obtaining various initial ullages,

4) Pressure and stroke calibration,

5) Burst diaphragm thickness.

Upon completion of the equipment calibration, emphasis was to be placed on arriving at conditions that would ensure 50% probability of reaction of 0.010 in. thick samples of a titanium alloy, either 5 Al - 2.5 Sn or 6 Al - 4 V, as directed by the contracting officer and of Dow Corning Oil FS 1281 both materials tested with liquid oxygen.

Various bubble volumes and rates of energy input per unit bubble volume in the oxidizer were to be set to determine if reaction occurred. A statistical analysis of the results were to be made and the results correlated with results furnished by NASA from the ABMA Drop Weight tester applied to the same material.

The parameters that were to be varied for the different engineering sample to be subjected to test were:

1) Thickness in inches of sample materials.

Titanium Alloy

a) 5 Al - 2.5 Sn: 0.005, 0.010, 0.025, 0.032, 0.063, 0.125, 0.250

b) 6 Al - 4 V: 0.005, 0.010, 0.025, 0.032, 0.063, 0.125, 0.250

Aluminum Alloy 5052-H-32

0.005, 0.010, 0.025, 0.032, 0.063, 0.125, 0.250

Magnesium

0.005, 0.010, 0.025, 0.032, 0.063, 0.125, 0.250

Narmco "C"

0.005, 0.010, 0.025, 0.050

FS 1281, 10,000 CS

0.005, 0.010, 0.025, 0.050

DC 33 Grease

0.005, 0.010, 0.025, 0.050

n-Hexane

0.005, 0.010, 0.025, 0.050

Bestoil

0.005, 0.010, 0.025, 0.050

2) Initial temperature (same as initial oxidizer temperature)

The results from each series of tests were to be statistically analyzed to determine confidence limits and be correlated with drop weight test results.

2.3 DELIVERY REQUIREMENTS

Upon the completion of the year's activity Airco was to deliver to NASA, Huntsville a fully equipped working prototype of the adiabatic compression apparatus along with operating and maintenance instructions. The amplifying and recording equipment for the instrumentation was not delivered with the rig since they belonged to Airco. This report satisfies these last requirements in addition to including a description of the equipment.

3. ADIABATIC COMPRESSION TESTER

3.1 MECHANICAL CONSTRUCTION

The basic design of this apparatus is built similarly to the standard piston apparatus as described and recommended for use by the Joint Army-Navy-Air Force Panel on Liquid Propellent Test Methods.

The unit constructed on this project was designed to provide a test chamber which can be rapidly pressurized by means of a pressure multiplier piston. Fig. 1 shows the final design of the apparatus. A complete set of drawings showing equipment details is included in Appendix B. The area ratio of the multiplier is 30:1 so that when the static pressure on the large piston is 1000 p.s.i.g., static pressure in the test chamber is 30,000 p.s.i.g. The test chamber itself is formed by the small end of the piston, the walls of the cylinder in which it moves, and a burst diaphragm held in place by a retainer. The cavity size of the test chamber has a volume of approx. 5 ml. and will accept both liquid and solid test material samples from 0.001 - 0.250 in. thick. Initial bubble volumes of 0.1 - 3.0 ml. can be achieved to permit obtaining a larger range of energy inputs necessary for establishing the sensitivity parameters of the experiment. The burst diaphragm is used to seal the chamber and preclude damage to the apparatus if the test chamber pressure substantially exceeds the design chamber pressure as a result of a positive reaction.

The burst diaphragm thickness was determined by using the formula:

$$\sigma_{max} = 3/4\ (qa^2/n^2)$$

where

σ_{max} = Yield point stress of material
q = Load/unit length intensity (p.s.i.)
a = Outer radius (sq.in.)
h = Thickness (in.)

This formula is based on the max shear theory where σ_{max} = 0.5 σ yield point in simple tension.

The test chamber portion of the apparatus was separated from the driver section and jacketed for two reasons as follows:

1) To minimize the heating or cooling time of the chamber by offering a much smaller mass of material to be affected.

2) To minimize the problem of sealing the larger piston statically and dynamically under cryogenic consitions. Axial clearance between the two housings was held to 0.0004 to 0.0005 in. to insure acceptable seal clearance in the test chamber. The operating temperature range of the tester is from +300°F to -300°F.

A detent pin has also been incorporated in the drive piston and consists of a lock rack and pin. The rack is adjustable to permit variation in compression strokes. Shims are inserted between the end of the rack and piston to prevent slippage when locking at the end of a stroke. This device is designed to prevent or limit piston bounce at the end of the piston stroke. It is recognized that as compression in the test chamber occurs, the volume of the oxidizer is heated, and the heat is transferred to the test material in contact with the oxidizer until a reaction temperature is reached. The material must be held at this temperature for a sufficiently long period of time for the reaction to continue. If piston bounce prevails, oscillation of the piston would cause a series of compressions and expansions within the sample. Such a phenomenon implies that the energy input level must be relatively high because the temperature the sample feels must be higher than if the sample were heated almost instantaneously and then held at that temperature.

A filling port is provided at the top of the test chamber for oxidizer loading. Its operation is fully explained under Section 3.4, Test Chamber Loading System.

In order to prevent the small piston from contacting the burst diaphragm at the end of a compression stroke, a shim spacer is calibrated, assembled and located into the larger piston cavity before final assembly. The shim consists of a Teflon insert in a brass housing. Final clearance should be checked just before inserting the burst diaphragm on an initial assembly to make sure the piston will not strike the diaphragm at the end of the stroke.

A considerable effort was expended in attaining a cryogenic seal to hold 30,000 p.s.i.g. at -300°F. Many seal manufacturers were contacted and a considerable amount of information attained. However, of all seals and seal configurations tried, an Omniseal Part No. EX 6802-2 manufactured by Aeroquip Corp., Burbank, Calif. was the only one which successfully fulfilled the low temperature, high pressure requirement. Only after adapting an entirely new seal configuration, as shown in Fig. 1, was sealing of the test chamber completely achieved. The design advantages of the new seal configuration are as follows:

1) The seal is stationary thus eliminating the drag of the seal along the face of the insert as experienced when the seal originally was mounted on the piston.

2) Being stationary, the seal is not exposed to damage occurring if it were necessary to pass over the filling hole or any sharp edge.

3) With the seal mounted in the outer housing insert, the seal shrinkage acts to advantage by increasing the interference between the seal and piston and thereby exerts greater seal potential.

3.2 MECHANICAL-PNEUMATIC SYSTEM CONSTRUCTION AND OPERATION

An assembly sketch, Fig. 2, shows the schematic diagram of the pneumatic system, Figs. 3 and 4, are photographic views of the tester assembled and mounted in the test cell. Visible in the pictures are some of the components of the pneumatic drive system; the activating control solenoid, a portion of the surge tank, the orifice retainer, and the temperature control system flow lines to the test jacket. The strain gage leads can be seen coming from the top of the rig and the piston displacement instrument mounted at the bottom of the rig.

The operation of this pneumatic system, in preparation for a test firing, is as follows: Gas (either nitrogen or helium) from high pressure supply is loaded into the drive gas storage tank, which has an approximate storage capacity of 97 s.c.f., to the desired operating pressure. This chamber was specified such that little or relatively no pressure drop was experienced in the driving chamber when being pressurized by the sudden opening of the control solenoid firing valve. This keeps a uniform unchanging pressure applied to the driving piston at all times during a firing. The pressure in the driving gas tank is read accurately using an Ashcraft Laboratory Test Gauge, Catalog No. 1279A, having an 8-1/2 in.diam. face and pressure rated from 0 - 1000 p.s.i. with 10 p.s.i. graduations. A Barksdale Type 42253AS2A1 solenoid valve is actuated to fire the tester. This valve has a nominal flow passage of 7/32 in. The orifice union located close to the solenoid valve, holds various orifices used for attaining different rates of pressure rise in the driving cylinder. After

several calibration tests, the length of the lines between the solenoid valve and orifice holder and the driving cylinder fitting were further shortened and straightened to minimize the effects of pressure drop in the line and the obtaining of the fastest rate of pressure rise in the test chamber. Any obstruction in this line would only slow the pressure response time. The photographs, Figs. 3 and 4, show the longer lines. Not shown in these figures is the Asco vent solenoid. The Asco valve is used in the chamber filling cycle to allow repositioning of the piston (placing a positive pressure greater than the test chamber pressure) to seal off the filling port after the filling operation is completed. Refer to Section 8.3 of this report for more detailed description.

3.3 INSTRUMENTATION

3.3.1 Description of Instruments

Instantaneous pressure in the test chamber is sensed by a Baldwin Lima Hamilton strain gage SR4 Type A-19 mounted on a burst diaphragm which is used to form one end of the test chamber. Satisfactory bonding of the gage to the diaphragm was obtained using Duco Cement for ambient temperature and Epoxy Cements EPY-150 for cryogenic temperatures. Standard BLH manufacturers recommended bonding procedures were followed when cementing gages to the disc. A second strain gage, which is attached to the rig retaining ring in a region close to the first diaphragm strain gage, is used to compensate for any change which might occur in the strain signal due solely to temperature change.

A Schaevity standard linear variable differential transformer, Type 500 HC, is used to measure piston displacement. The feature of this unit eliminates direct contact of any parts and renders the parts impervious to most environmental extremes. The electrical output of this infinite resolution device is proportional to the displacement of a "floating," movable core and can be calibrated and used to indicate distance or linear displacement.

The two types of external transducers (strain gage and linear variable differential transformer) were used in conjunction with two Sanborn Strain Gage Amplifiers Model #64-500A, one for each transducer. In designing the instrumentation the transducer was assumed to be a simple Wheatstone bridge and excited by a 2,500 cycle oscillation. The output terminals of the bridge were connected to the carrier amplifiers through a matching transformer and step attenuator. When the bridge is perfectly balanced, no voltage will be delivered to the carrier amplifier. If, however, one of the arms of the bridge should change in resistance, the balance will be destroyed and some fraction of the excitation voltage will reach the carrier amplifier. The magnitude of this unbalance voltage depends upon the magnitude of the resistance change, while the phase of the unbalance voltage depends upon whether the resistance change was an increase or a decrease from the initial balance value.

In order to attain sufficient galvanometer displacement on a Heiland Visicorder Model 906B Oscillographic Recorder, the output signal from the excited Sanborn amplifier was directed through a galvometer driver, Fig. 5b, before entering the recorder. Two Heiland Model M-3300 Galvometers were used in the recorder. Paper speed of 50 i.p.m. was found most satisfactory in recording piston displacement. Fig. 5a shows the schematic instrumentation hookup while Fig. 6 shows the mounting of the power supply and amplifier.

A time reference recording of 60 cycle a.c. was also found most useful in data reduction of the traces.

All of the amplifying and recording equipment used at the Airco Murray Hill Laboratories was not procured under this contract but was drawn for use from the inventory of available instrumentation. Accordingly, it was not supplied to NASA with the remainder of the test apparatus. For duplication of the functions of the amplifying equipment it is recommended that any carrier amplifier with an exciting frequency of 2.5 kc. used in conjunction with a recorder capability of a 1000 cycle band width can be used with any one of the two transducers.

Attempts to obtain a thermocouple which might support a positive conclusion as to whether or not a reaction due to volumetric heating had occurred in the test chamber proved unsuccessful. Thermocouples with a response rate rapid enough to give us any reasonable assurance could not be found. The only equipment which can respond to this rate of temperature change would be a radiometer device which would require an optical path to view the test cavity directly.

3.3.2 Operation of Recording Instrumentation

The following sequence of operations is recommended:

1) Amplifier and recording instruments are warmed up for 30 min.

2) The linear differential transformer is calibrated measuring the actual displacement versus the spot deflection on the oscillograph.

3) The test chamber is loaded and sealed as described in Section 3.4, Chamber Loading System.

4) The desired driving gas pressure is imposed in the surge reservoir.

5) A sufficient period of time (normally 15 min.) is held to insure thermal equilibrium.

6) The firing button is closed. This initiates a timer sequence which starts the oscillograph film drive and allows the drive mechanism to come up to speed. A 60-cycle trace is superimposed on the oscillograph film to give a more accurate estimate of the time.

7) A mechanically timed switch then activates the solenoid valves and allows the high pressure gas stored in the storage tank to pressurize the driving chamber of the tester. The piston is driven forward into the test chamber and the linear displacement of the piston recorded.

8) A second timer releases only the Barksdale solenoid valve allowing the driving chamber to vent through the Asco solenoid valve.

9) A third timer cuts the power to the oscillograph drive and the Asco valve is reset.

10) If no explosion has occurred the piston is retracted, the test chamber opened and its contents examined for evidence of decomposition.

11) If an explosion occurs, the test chamber is opened and examined for possible damage. The complete test section housing is removed from the rig and completely disassembled, except for the refrigeration jacket. All parts are thorcughly cleaned of the decomposed residue and reassembled for further tests.

12) Exposed oscillograph film is removed and examined and the piston velocity directly determined from the trace of the piston displacement.

3.4 CHAMBER LOADING SYSTEM

In the case of charging liquid oxidizers at ambient temperature, the tester is placed in an upright position so that the test chamber section forms a cup. Note: The diaphragm and retainer nut have not as yet been assembled to the rig. The piston is hand-positioned by means of a depth micrometer so that the desired liquid volume for the test will completely fill the chamber. The chamber is then sealed with the burst disc diaphragm and the desired bubble volume formed by retracting the piston a

predetermined measured amount. Any relationship between a liquid volume and bubble volume can be obtained in this manner as long as the total volume does not exceed 5 ml.

In the case of charging gases to the chamber, a filling port opening is provided which allows the chamber to be filled after the cavity has been sealed. Prior to actually charging, the chamber must be purged with the gas to effectively remove air from the chamber. Calculations indicate, assuming complete mixing, that 3 - 4 pressurizations with oxygen gas at 2000 p.s.i. and evacuations will effectively remove all residual nitrogen. The piston cavity is then charged to give the desired quantity of gas in the chamber. Likewise subsequent movement of the piston will give the desired pressure variations. In this procedure the initial minimum volume is limited in that the piston must be retracted to a position allowing the charging port to be exposed to the chamber.

After charging the chamber, the piston is repositioned to cover the charging port and the temperature of the rig is raised or lowered to the desired level. As the rigs temperature is lowered to the point of condensation, a known quantity of liquid will be formed in the test cavity.

4. TEMPERATURE CONTROL SYSTEM AND OPERATION

The temperature control system for the adiabatic compression tester is composed of four individual systems as shown in flow diagram Fig. 10. They provide ability to obtain a constant temperature environment for the test rig from -300°F to 300°F at any preset temperature. The four systems are:

		Temperature Range
1)	Liquid Nitrogen	-300 to -290
2)	Freon-13	-290 to -150
3)	Freon-11	-150 to +60
4)	Water	+60 to +300

Figs. 8 and 9 show the installation of the system at our test facility.

4.1 LIQUID NITROGEN COOLING SYSTEM

4.1.1 Storage

The liquid nitrogen is stored in a conventional industrial liquid nitrogen customer station which provides its own safety provisions and the means for maintaining sufficient pressure to allow continuous flow of liquid. Fig. 10 is a schematic flow diagram of this phase of the system.

It should be noted that the LN_2 supply station was not shipped with the remainder of the equipment inasmuch as it was not procured under this contract.

4.1.2 Fluid Flow Temperature Control

Liquid nitrogen is piped to the test rig in insulated lines to minimize heat gain from the surroundings. Liquid nitrogen is also used as the coolant in both Freon-11 and Freon-13 heat exchangers. At the exit of the rig a back pressure is maintained controlling the boiling temperature. A pressure regulated control valve maintains the required back pressure. Nitrogen gas is vented to the atmosphere.

4.1.3 Safety Considerations

All lines are protected by relief valves. The trapping of liquid between valves should always be avoided.

4.1.4 Operation of Liquid Nitrogen Cooling System

Liquid nitrogen is pressure fed to the system under a head of 40 - 50 p.s.i.g. from the customer station. This pressure head is maintained at the station by manual operation of

appropriate valving permitting flow through the pressure build-up coil. The liquid take-off valve (400)* is then opened placing the station on stream. The solenoid valve (401) is placed in the full open (manual switch position). Valves 402 and 601 are opened allowing entrance to the test rig; all other valves should be closed. At the exit of the test rig, the flow of nitrogen is initially vented through the control section by pass valve (602) and nitrogen exhaust valve (603). After this initial cool down of the test rig and lines, the back pressure control section is placed in parallel (valves 411 and 414 are opened) air supply to pressure valve (413) and pressure controller (PC-1) is turned on, the by-pass valve (602) is closed. The controller is adjusted to give the required back pressure at the rig thereby regulating the boiling point of the liquid nitrogen in the test rig and thereby the temperature. Approximately 45 - 60 min. are required after the temperature control system has been set to the desired temperature, for the test rig to reach thermal equilibrium.

4.1.5 Shut Down

Normal shut-down procedures require the valve at the nitrogen station be closed and the lines vented. The air supply to the control valve is then shut off.

4.2 FREON-13 COOLING SYSTEM

4.2.1 Storage Tank

The storage tank consists of a heavy walled stainless steel tank, ASME Code pressure rated at 1000 p.s.i. to permit storage of Freon-13 at room temperature. The tank is sealed from connecting piping during storage by high pressure valves. A heat exchanger coil is incorporated within the tank as are both temperature measurement and liquid level probes. During operation of the adiabatic tester liquid Freon-13 (below -114°F) is pumped from the storage tank through the test rig and returned to the storage tank. Fig. 11 shows a schematic flow diagram of the F-13 phase of the system†.

In the event of a rupture or leak in the system's lines the piping is equipped with stop-flow features to prevent the loss of the contents of the storage tank. A pressure activated control valve seals the outlet of the storage tank at the suction of the pump and a check valve prevents reversal of flow and the resulting loss at the return inlet of the tank.

4.2.2 Charging the Storage Tank

Since Freon-13 is a gas at room temperature, it is necessary to condense the gas while charging from the compressed gas cylinders. During charging and storage the storage tank is

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* For valve numbers refer to Fig. 10.

† For valve numbers refer to Fig. 11.

sealed from the remainder of the system by the high pressure valves (301, 314 and 311). The storage tank is charged through the fill valve (316). Condensing is accomplished by using the heat exchange coil within the F-13 storage tank. Liquid nitrogen from the customer state is used as the cooling medium. Initial cool-down is accomplished by opening valves 400, 401, 403, 410, by-pass valve 407, and vent valve 409. Once the entire system has been cooled down, throttling is accomplished by closing 410 and opening valves 415 and 416. Various temperatures are obtained by the pressure controller on the output line of the heat exchanger. Valve 407 is closed and valve 405, 406 and 408 are put into operation.

4.2.3 Operation of the Freon-13 Cooling System

Before attempting to use Freon-13 in the system, the contents of the storage tank must be brought below the atmospheric boiling point of the liquid (-114°F) and all lines cooled with liquid nitrogen to prevent excessive flashing of the Freon when the tank is opened. Liquid nitrogen is admitted to the system (valve 313) and passed through the system venting at valves 607, 315 and 302. The storage tank is opened to the system and the pump is filled by gravity feed, venting the storage tank at valve 607. In order to do this, the pressure activated control valve (305) is by-passed (valve 304) since the control valve is closed unless a minimum pressure is maintained at the pump suction. The tank vent valve (607) is closed as is the control valve by-pass (304). The pump is started, circulating Freon-13 through the system.

4.2.4 Cryogenic Pump

The cryogenic pump used is a Paul Pump requiring a minimum of 10 p.s.i.g. suction pressure at the pump suction connection. The pump seal is protected by relief valves. The pump is started and a back pressure is maintained at the pump discharge by regulating the needle valve (606) and the liquid return line valve (312)[209 F-11]. (NOTE: Follow manufacturers instructions when operating pump at various temperatures. Instructions have already been sent to NASA.)

4.2.5 Shut Down

1) The pump is shut off.

2) The pressure activated control valve (305) is by-passed.

3) The outlet of the storage tank is closed (valve 301).

4) The contents of the lines are blown back into the storage tank using nitrogen gas (valve 303).

5) The storage tank is sealed off from the system by closing valves 314 and 311.

6) Lines are vented to prevent excess pressure build-up valves 315, 607, 603, and 302.

4.3 FREON-11 COOLING SYSTEM

4.3.1 Storage Tank

The storage tank for Freon-11 is a 65 gal. stainless steel tank pressure rated at 150 p.s.i. Liquid level readings are taken using a dip stick. The tank is protected by a relief valve (S-28). Freon-11, a liquid, is charged into the tank until the desired quantity is obtained through valve 207 while venting (valve 208). At ambient temperature and pressure Freon-11 is a liquid. Fig. 12 shows a schematic flow diagram of the Freon-11 phase of the system*.

4.3.2 Operation of the Freon-11 Cooling System

Initially the storage tank is pressurized to approx. 10 p.s.i. With gaseous nitrogen (206), the outlet valves 210, 201, 304 and 306 are opened and the liquid allowed to fill the F-11 heat exchanger. Valves 202 and 204 are open while venting (valve 203). This will also fill the pump (P-1) with liquid. The pressure in the tank is relieved (valve 208) and the return line valves (205 and 209) are opened. The throttle valves 606 and 605 are opened. Valve 605 and valve 309 permit the flow of liquid to by-pass the heat exchanger.

The pump is started and the throttle valve (606) is adjusted to give the required back pressure at the discharge of the pump (85 p.s.i.) allowing some flow back to the tank through valve 209. The remainder of the flow is routed either around or through the heat exchanger (F-11), to the test rig and returned to the storage tank.

Liquid or gaseous nitrogen is supplied to the heat exchanger from the liquid nitrogen station. Control of the flow of nitrogen is maintained by use of a temperature controller (TC-1) which operates a solenoid valve (401).

The temperature at the rig is controlled by adjusting both the flow rate through the heat exchanger and the controller (TC-1) set point.

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* For valve numbers refer to Fig. 12.

4.3.3 Cryogenic Pump

See Section 4.2.4 for operation of the Freon pump.

The pressure regulated control valve (305) designed to prevent the loss of the contents of the storage tank when using Freon-13 may also be used in the Freon-11 system following the procedure outlined under Freon-13 operation instructions.

4.3.4 Shut Down

1) The pump (P-1) is shut off, by-pass (305) is opened if used.

2) The outlet of the storage tank is closed (valve 210).

3) The contents of the lines are blown back into the storage tank using nitrogen gas (valve 303).

4) The storage tank is sealed off from the system (valves 206 and 209).

5) Lines are vented to prevent excess pressure build-up (valves 302 and 607).

4.4 WATER HEATING SYSTEM

4.4.1 Storage Tank

The storage tank for water is a 65 gal. stainless steel tank pressure rated at 150 p.s.i. The tank is protected by a relief valve (S-3). Water is charged to the tank through valve 109*. Liquid level readings are taken using the liquid sight glass valves 110 and 111. During normal operation these valves (110 and 111) seal the sight glass from the tank. Fig. 13 presents the flow diagram for this phase of the system.

4.4.2 Operation of the Water System

Initially a pressure head is built up in the storage tank (20 p.s.i.). The outlet valve is opened (101) bleeding the lines (valve 603) in order to fill the pump, heater and lines with liquid. The tank is then vented (107) and the pump is started, circulating the water through the test rig and back to the storage tank. The temperature controller on the line water heater (H-1) is set to provide the desired temperature at the rig. Operation instructions of the water pump and heater have already been furnished to NASA.

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* For valve numbers refer to Fig. 13.

4.4.3 Line Water Heater

The heating apparatus of the water system consists of an electric heater combined with its own temperature indicator, and controller.

4.4.4 Shut Down

1) The power to the heat exchanger (H-1) is cut off.

2) The pump (P-2) is shut off.

3) When operating under pressure the system is vented slowly to the atmosphere.

4) The outlet of storage tank is closed (101) and lines blown clear of liquid back to the storage tank (valve 104).

5) The storage tank is sealed off (valve 106).

5.1 STATIC CALIBRATION

Actual calibration of the piston displacement was accomplished by using the variable transformer, which is attached to the drive piston assembly, and measuring the actual piston displacement with a vernier caliper. The results were recorded and noted on a Visicord oscillograph paper. For our calibration, a total stroke of 0.738 was recorded on the trace paper. For every 0.10 in. gradient line on the trace paper, a piston movement of 0.01497 in. was represented. This information was most useful in determining bubble volume when loading the test chamber with a fuel sample to be tested. The volume change with piston stroke is such that for every incremental 0.01497 in. change in stroke, a corresponding volume change of 0.1 ml. occurs in the test chamber.

The test chamber pressure was calibrated using the two Baldwin Lima Hamilton strain gages, Model No. SR4. One unit was attached to a 1/4 in. thick hardened stainless steel burst disc which forms one end of the test cavity. The other unit was attached to the retainer ring and used strictly as a temperature compensator. The gage on the diaphragm was calibrated by statically loading the test chamber with driving pressures in the driving chamber from 100 - 1000 p.s.i. and recording the strain gages output on the oscillograph. The results of these calibrations are shown in Fig. 14. Also shown on the same figure is the driving pressure relation to the test chamber pressure; this last relation is plotted for more readily interpretation in Fig. 15.

5.2 DYNAMIC CALIBRATION

The rate of pressure rise in the test chamber was measured with the test chamber filled with a liquid so that the piston did not move. Smooth pressure traces were obtained with the ranges of pressure rise rates measured in the test chamber from 299×10^3 to 1708×10^3 p.s.i./sec. as shown in Table 1.

Variations from the particular rate of pressure rise were readily obtained by variations in the driving gas and changing the orifice size between the high pressure surge tank and the driving chamber of the rig.

With the assumption confirmed that the pressure rise in the test chamber is very rapid, the compression will be nearly adiabatic thus resulting in a rapid temperature rise. Therefore, the assumption that the sensitivity of the test materials in the presence of oxygen is being tested in the presence of a very rapid temperature increase can be made.

In estimating the energy input to the test sample it is proposed that the gas expanding from the reservoir transmits kinetic energy to the piston and this kinetic energy of the piston is expended in compressing the gas in the test chamber. Then the energy expended on the compression of the gas in the test chamber would be primarily based on the mass of the piston and the velocity of the piston. The mass of weight of our piston is 3283 g.

5.3 TESTER CHARACTERISTIC CALIBRATION USING n-PROPYL NITRATE

To aid in establishing the tester's characteristics and to compare its performance as well as its ability to produce data which has been already documented, several tests were run with n-propyl nitrate in the presence of two different bubble volumes. These data are shown in Table 2.

Based on these few tests plus the knowledge that previous NPN sensitivity data was based on results which were the average of many tests and whose energy levels were in some cases as low as 3.3 kg-cm. for positive results and negative results as high as 5.9 kg-cm., it appeared that we had obtained sensitivity results somewhat higher than others reported. This may be attributed to our upward firing position, putting the bubble in contact with the aluminum burst diaphragm rather than the bubble in contact with the piston system. There is also the question as to the consistency of the bubble. It is understood that ignition of the fuels is less sensitive in the presence of a vapor bubble than in an all air bubble or fuel vapor - air mixture.

Another significant result of these tests is that the tester maintained mechanical integrity during firing. There was some concern about this since previously built testers having roughly the same mechanical constraint contained only about 30% of the propellant mass as has the current design.

The locked stroke device calibration was not made during the course of the work. The locking pin and rocket positioning can be established from knowing the maximum stroke length determined prior to the test (refer to Section 8.2).

6. MATERIAL EVALUATION TESTS

The only material to be tested was on 0.010 in. thick titanium disc (5 Al - 2.5 Sn) in the presence of 0.77 ml. of gaseous oxygen at ambient temperatures. The titanium disc was positioned just in front of the aluminum rupture disc (0.305 in. thick). This particular disc was calculated to yield at 11,565 p.s.i. chamber pressure.

On the first series of tests a precautious approach was taken. Six tests ranging from 100 p.s.i.g. drive pressure of helium gas with no orifice, to 300 p.s.i.g. drive pressure at 50 p.s.i.g. increments were taken with negative results. At this point the aluminum burst diaphragm was replaced with one (0.372 in. thick) which would yield at 17,700 p.s.i. chamber pressure. This permitted the extension of the driving pressure range to be increased to 525 p.s.i.g. A repeat test at 300 p.s.i.g. resulted in a negative test. The drive pressure was then increased to 350 p.s.i.g. and a bright flash was noted. On observing the rig, it was noted that the burst diaphragm had blown out. The rig was disassembled, with some difficulty, and the results are shown in Fig. 16. The titanium disc had burned to a complete circle of 0.75 in. which is the test chamber diameter. The aluminum burst disc had melted and blown out; and the stainless steel insert and piston had also melted in the immediate area of the test chamber area. This of course had caused the problem in disassembling the rig.

The recorded data indicated that the piston had traveled a distance of 0.489 in. in 0.0045 sec. indicating a piston velocity of 266 cm./sec. Based on these data the energy input to the bubble was calculated to be 118.2 kg. The energy input to the negative test at 300 p.s.i.g. was 44.4 kg-cm.

At this point three revisions were incorporated in the tester in order to eliminate the damage to the insert and piston. One was to modify the piston to incorporate an inexpensive removable tip. The tip, if damaged by a positive test, can be readily replaced at low cost. The second modification was to reduce the area of the titanium sample disc so that it would not come in direct contact with the stainless steel insert. Therefore, when a reaction occurred and the titanium started to react and burn, it would not possibly burn to the edge of the insert causing the stainless steel to react and melt.

This second modification was accomplished by placing a stainless steel washer, notched to receive a 1/2 in.diam. test sample and placed at the end of the test chamber directly in and against the face of the burst disc.

The third modification was to drill a small relief hole in the rupture diaphragm in the center directly behind the test sample and almost through the disc. Structural modifications were also made to the burst disc to compensate for the drilled hole. It was anticipated that if the titanium reacted, burning of the titanium

in the center of the sample would melt the aluminum at this thinned section and provide a guided pressure relief point so that reaction would not proceed further and thereby limit burning damage to the rig. Fig. 17 shows the modified sample retainer in the test chamber.

With the completion and incorporation of the above changes, a series of tests were again performed at ambient temperature with gaseous oxygen. A repeat of the 300 p.s.i.g. driving pressure test indicated a negative test. The driving pressure was then increased gradually until a positive test was achieved at 900 p.s.i.g. drive pressure. The reaction occurred with the results as shown in Fig. 15. The small 1/2 in.diam. titanium disc was completely burned. The stainless steel holding washer was damaged on one side and the burst disc was also burned or melted on one side. Unfortunately the melting of the burned disc had not reached the drilled relief hole and the reaction proceeded between the burst disc face and the insert face in the vicinity of the flange seal and finally up the side of the retaining insert and tester housing. Considerable damage was inflicted on the test housing and retainer ring as shown in Fig. 19. However, all parts were reusable after some light rework and cleanings.

The pressure relief hold was deepened and enlarged in the burst diagram and again with compensation given to its structural strength. Another test was run under basically similar conditions as the previous above test. A positive result was experienced at 750 p.s.i.g. driving pressure with the results as shown in Fig. 20. Ignition of the aluminum rupture diaphragm occurred at the edge of the test chamber and again the reaction proceeded between the face of the diaphragm and the piston insert in the area of the "O" ring seal. The titanium test sample did not react as expected as evident in the picture. Slight burning did occur at the edge of the holding washer and across the face of the piston insert.

A review of the three positive tests indicate that possibly Tests 2 and 3 were the results of a reaction between the oxygen and aluminum without a reaction of the titanium test sample. In Test 1 the aluminum burst disc might have started reacting and progressed to the titanium. There also exists the possibility that the "O" ring between the insert and burst disc might have leaked in Test 3 starting the reaction between the aluminum and the high flow rate of the oxygen.

To review and explain what might occur with reactions of metal in the presence of oxidizers, it is commonly supposed that of metals which form protective oxidizers ignition will occur only after the protective oxide becomes molten or after the metal is broken in such a way as to expose a nascent surface to the hot oxidizing atmosphere. It is also felt that a metal will react and ignite at a low temperature if it is not prevented from doing this by an impervious protective oxide. Thin and weak but impervious oxide coatings will break under pressures slightly above ambient pressures. If the oxide coating is thin and permeable, sufficient metal vapor will diffuse through the coating to cause ignition in the vapor phase external to the unbroken oxide coating. For oxide coatings which are thick, strong, and impervious, a significantly higher than

ambient vapor pressure will be needed to break the coating. Such factors as these must be weighed in explaining the causes of a positive reaction.

Indications are that the positive results we attained during these tests might be caused by the breaking of the oxide coating mechanically. Because of the holding method of the test sample and burst disc (reclamping between the piston insert and retainer ring) oxide failure could be occurring at the edge of the test chamber around the outer circumference of the test sample where the maximum and sharpest deflection occurs when the test chamber is pressurized. With the reduction in size of the titanium sample disc, it is no longer subject to the sharp deflector area and may assume the softer contour of the deflecting aluminum burst diaphragm. In the Positive Tests 2 and 3, the aluminum burst diaphragm was subject to the sharper and maximum deflection at the supporting edges of the chamber which could cause its oxide coating to rupture, thereby causing reaction. It is concluded that from the three observed positive results obtained, Test 1 was a definite positive reaction between the oxidizer and the titanium. However, some doubt does exist as to what started the reaction in Test 2, the aluminum burst diaphragm or the titanium sample disc. Test 3 indicates that the aluminum reacted with no reaction of the titanium sample.

It must also be realized that when a reaction between an oxidizer and test sample occurs within the test chamber, the burst diaphragm may not always rupture. A reaction occurring in the test chamber ordinarily consumes the oxidizer with a corresponding increase in temperature of the gas. When the test chamber contains only a relatively small amount of oxidizer per unit volume, it may only be sufficient enough to support a reaction for a short period of time after initiation, therefore as the reaction occurs, the oxidizer is consumed with a resultant decrease in chamber pressure. The only damage inflicted on the diaphragm may be melting of the disc due to the increase in chamber temperature.

When consideration is given to a reaction occurring with more than sufficient oxidizer present to support a reaction, i.e. as a liquid or high pressure gas thus having a greater mole density per unit volume, there is enough unconsumed gas present in the chamber to expand with the increase in temperature after initiation. Therefore with the rapid gas expansion the resultant increase in pressure will eventually cause diaphragm distortion or rupture.

Because of the short time remaining before the termination of this contract, the experimental work was halted in order to emphasize the attaining of the final step under the testers physical requirement. (The apparatus should operate from -297.4°F to 302°F and provide a maximum driving pressure to give a maximum static pressure in the sample chamber of approx. 30,000 p.s.i.). At this time the above conditions had been met except for a step from 24,000 p.s.i. to 30,000 p.s.i. at -300°F. The reason for the shift

was to be sure to provide NASA with a tester which would not only allow them to test engineering materials in the presence of liquid oxygen but would provide sufficient pressure to insure 50% probability of reaction of 0.010 in. thick samples of titanium alloy in liquid oxygen. This revision of work was also agreed to by NASA on a visit to Air Reduction by Mr. F. Key in April 1966.

7. ANALYSIS OF ROUTINE TESTING PROGRAM

7.1 REQUIREMENTS

A review of the number of variables to be considered in a routine adiabatic compression testing program has indicated that an extremely large number of test determinations are required. Accordingly a statistical experimental approach* has been devised and is recommended for use with the tester.

In the program scope, the following variables are encountered:

Oxidizers	3	
Materials	9	Species 48
Thicknesses	7,4	
Position of driving piston	3	
Rate of compression	X	
Quantity of gas compressed	Y	
Initial pressure	Y	
Initial volume	Y	
Initial temperature	Z	

Total number of points

$$3 \cdot 48 \cdot 3 \cdot X \cdot Y \cdot Z = 432\ XYZ$$

7.2 ANALYSIS OF EFFECTS

Assume any values for X, Y, and Z which will give the total number of points to be investigated, for example:

If

$$X = 5$$
$$Y = 5$$
$$Z = 5$$
$$XYZ = 125$$
$$\text{Total points} = (423)(125)$$
$$= 52{,}875$$

---

* DIXON, W.J. and MASSEY, F.J.
Introduction to statistical analysis.
New York, McGraw Hill, 2nd ed., 1957.

VILLARS, D.S.
Statistical design and analysis of experiments for development research.
Dubuque, Iowa, Wm. C. Brown Co., 1951.

If

$$X = 5$$
$$Y = 2$$
$$Z = 1$$
$$XYZ = 10$$
$$\text{Total points} = (423)(10)$$
$$= 4,230$$

The analysis of the effects of these variables on the energy input to cause reaction can be presented in terms of a critical energy input. The critical energy input associated with each specimen is defined as that energy above which the specimen statistically will always react with the oxidizer. In sensitivity testing, the specimens are characterized by a continuous variable (the critical energy input), which cannot be determined directly as a discrete point. Rather, measurements are made of energy input and determinations of sensitivity or lack thereof made above or below the critical value.

The technique of investigation involves the use of the "up-and-down" method which has been developed and used in explosives research. The advantage of this technique is that it concentrates testing near the mean which increases the accuracy with which the mean may be estimated. This results in a reduction in the number of observations of between 30 - 40%.

The number of tests required to establish the mean is dependent upon the spread, or variability, associated with the specimen. The choice of testing interval also affects the number of tests required by effecting the precision of the mean. Previous investigations involving impact testing have shown that approx. 20 tests are required to accurately estimate the mean, when testing intervals were of the magnitude of 0.5 - 2.0 of the standard deviation. Tests in excess of 20 were shown to have little effect on the mean, whereas below 20 tests, the effect of an additional test is large.

It was estimated that 20 tests will be required to accurately determine the mean. It is important to note that the "up-and-down" method of analysis is based on the assumption that the variate under examination be normally distributed. It is rarely the case that a natural variate is found to be normal. A transformation of the results into a normally distributed variate may be found to be necessary. In explosion testing this has been found to be a log transformation.

With this method of statistically analyzing the test results, a design of the overall experiment was examined.

7.3 QUALITATIVE EXPERIMENTAL DESIGN

Our estimate of the number of tests which can be conducted per day varies between a conservative estimate of 2 tests per day to a liberal estimate of 6 tests per day.

For example, in a 20 wk. testing period (100 days) the number of tests which can be run is estimated at 200 - 600 tests.

Assuming 20 tests to establish a point, 10 - 30 points may be covered. Knowing this, a specific material, material thickness, oxidizer and test position must be fixed. Initially it is recommended that titanium of 0.010 in. thickness be tested with liquid oxygen and the tester activated in an upstroke position (this position being selected to ensure that the engineering material is in contact with the gas bubble when a liquid is present). The literature suggests that this is where the interaction takes place.

With those parameters fixed, the rate of compression, quantity of gas compressed and initial temperature can be viewed. The number of levels of each respective variable can be chosen to conform to the number of tests which will be performed. It is felt that a minimum of 240 tests can be made. At 20 tests per point 12 points could be examined.

Our approach to the examination of these 12 points involves a complete factorial statistical design. The design incorporated 3 levels of temperature, 2 levels of quantity of gas compressed and 2 levels of rate of compression. This method of design yields the greatest amount of information concerning the main treatment effects and any interactions of all the designs with which we are familiar and that are applicable. Sampling of a larger factorial design results in a loss in total information obtained.

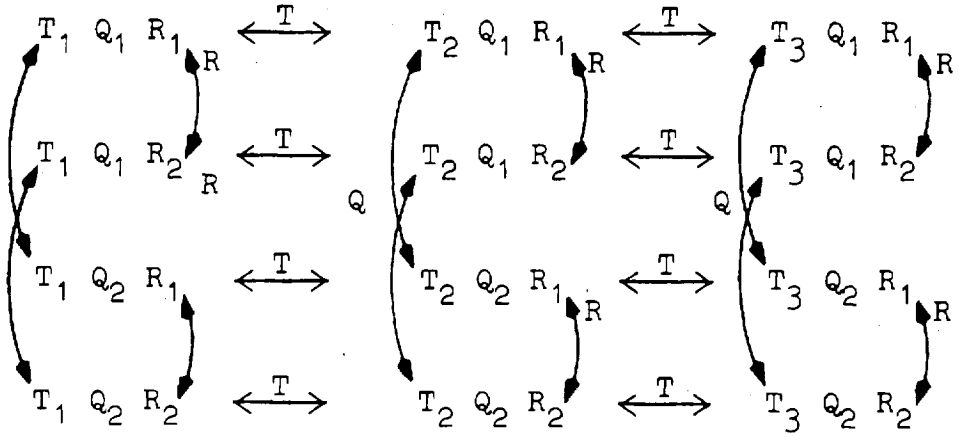
The factorial design additionally involves an analysis of variance by appropriate procedures to establish treatment differences with corresponding confidence intervals.

An example of the complete factorial design follows.

7.4 FACTORIAL DESIGN OF EXPERIMENT

		12 points
$T_2\ T_1\ T_2\ T_3$	(3)	Initial temperature
$Q_2\ Q_1\ Q_2$	(2)	Quantity of gas
$R_2\ R_1\ R_2$	(2)	Rate of compression

TEST DESIGN CONDITION (Test Points I thru XII)



$T_2 - T_1$ at 2 levels of Q, at 2 levels of R } 4

$T_3 - T_1$ at 2 levels of Q, at 2 levels of R } 4

$T_3 - T_2$ at 2 levels of Q, at 2 levels of R } 4

$R_2 - R_1$ at 3 levels of T, at 2 levels of Q } 6

$Q_2 - Q_1$ at 3 levels of T, at 2 levels of R } 6

The calibration tests can be made using an inert liquid to determine the time-temperature relationship in the compressed bubble without reaction. Instantaneous pressure and pressure rise in the test chamber can be determined by varying

1) Driving gas pressure,
2) Changing orifice size, and
3) Using different gases.

Also in determining the energy input, piston displacement is calibrated.

To estimate the critical energy input, a series of 20 tests should be performed using liquid oxygen and titanium. This will serve to ensure the 50% probability of reaction of 0.010 in. thick titanium alloy in the liquid oxygen.

It is suggested that the same statistical approach be used toward the evaluation of each of the other materials with each of the oxidizers.

8. EXAMPLE OF RIGS OPERATIONAL PROCEDURE

8.1 RECOMMENDED PROCEDURE FOR RIG ASSEMBLY (Refer to Fig. 1)

1) Assemble part C-898-5464, drive piston, to part A-898-5468, test piston.

2) Place the above assembly into housing C-898-5461 and assemble cover part C-898-5462 to housing.

3) Assemble insert A-898-5754 with A-898-5756 including seals and fitting #100-1-1-316 with 1/8 in. stainless steel tubing (1 ft. long).

4) Place this assembly into housing C-898-5463 leading the tubing through the hole provided in the housing.

5) Assemble housing C-898-5463 with jacket D-898-5460 to housing assembly C-898-5461 guiding the piston through the inserts very carefully. Bolt with spacers A-898-5472 between the housings.

6) Measure the thickness of the retainer nut B-898-5757 between the burst disc face and the outer face of the retainer.

7) Place the retainer nut in place and fully extend the piston into the inserts. Bolt the retainer in place and torque these bolts to 45 ft-lb.

8) Measure the distance between the outer face of the retainer nut and the end of the fully extended piston face.

9) From this measurement and the thickness measurement of the retainer nut, the clearance between the diaphragm face (when assembled into the rig) and the fully extended piston stroke can be calculated. This clearance is critical so that when the rig is fired the piston will not ram the diaphragm.

10) Compute the dimension of a mechanical stop spacer to give the desired clearance and insert this spacer into the upper housing C-898-5461 between the drive piston C-898-5464 and housing on the exhaust side of the drive piston.

11) After inserting the clearance spacer and reassembling the rig, recheck the clearance between the diaphragm and piston.

12) If acceptable, mount the rig in the test cell area and hook up all support lines (instrumentation, pneumatic and temperature control systems).

8.2 RECOMMENDED PROCEDURE FOR PISTON DISPLACEMENT CALIBRATION

1) Attach the linear differential transformer to the large piston as shown in Fig. 2 and hook up to the recording equipment.

2) Place the piston approximately at the midpoint of the piston stroke and adjust the transformer coil to obtain an approximate electrical equilibrium on the output signal of the secondary coils. (Use a voltimeter for this check.)

3) Null balance the transformer signal on the recording instrument.

4) With a vernier caliper or depth micrometer measure the actual piston displacement by hand moving the piston and relating the measured movements to the recording chart.

8.3 RECOMMENDED PROCEDURE FOR LOADING AND FIRING THE RIG USING AS AN EXAMPLE TEST GASEOUS OXYGEN AT 80°F AND A TITANIUM TEST SAMPLE

1) Assemble rig and place in testing cell.

2) Place titanium test sample in place so that it faces the test chamber. Back it with a burst diaphragm, if necessary, and assemble retainer and torque bolts.

3) Bring rig's temperature to 80°F using the water temperature control system; the temperature should be at equilibrium for at least 1 hr. after reaching the desired test temperature.

4) Warm up the instrument recorders for at least 30 min.

5) Purge the test chamber with oxygen through the filling line. (Be sure the piston is retracted far enough to uncover the filling port inside the chamber.)

6) Check the linear differential transformer calibration.

7) Fill the chamber with the desired quantity of oxygen. (Since the volume of the chamber is known, the quantity of gas can be determined by the gas temperature and pressure.)

8) Allow 15 min. for rig's thermal equilibrium.

9) Place the piston in the firing position. This is accomplished by moving the piston to close off the filling port. The piston is moved by pressurizing the driving piston chamber with the auxiliary gas supply. After proper positioning, close off gas supply from the auxiliary supply source.

10) Load the drive gas storage tank to the desired test pressure.

11) The firing button is depressed starting the recording instruments with the Barksdale valve opening after a short delay. The piston is driven forward into the test chamber, and the linear piston displacement is recorded.

12) The Barksdale valve then closes by a second timer which vents the drive chamber through the Asco valve which is opened at the same time.

13) The instrument recorders are turned off.

14) The Asco vent valve is then closed.

15) The rig is examined for reaction, and the recorder traces are developed.

9. INSTRUMENTATION SUMMARY LIST

1) Strain Gage: Baldwin Lima Hamilton (BLH) Strain Gage Cat. No. SR4-A19. Typical current carrying capacity of this gage is generally limited to 25 ma. The gage resistance is approx. 60 ohms ± 1.5 ohm spread. Its grid dimensions are 1/16 in. long × 1/16 in. wide.

2) Strain Gage Amplifier*: Sanborn Company Model 64-500 having an impedence under normal load of 3000 ohms. The maximum output is 25 ma. from center with a carrier frequency of 2500 cycles per second.

3) Linear Variable Differential Transformer: Schaevity Engineering Company Model 500HS having a linear range of 0.500 - 0 - 0.500 in. It has a linear accuracy of 0.5% of full scale. The excitation input voltage is 6.3 volts at 60 cycles per second.

4) Recorder: Honeywell "Visicorder" Model 906B with 14 channels. It records frequencies through a range of 0 - 3000 cycles per second. The trace may overlap permitting a full 6 in. peak to peak amplitude of recording. Damping resistors can be provided for each recording galvanometer channel.

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* Any carrier amplifier with an exciting frequency of 2.5 kc used in conjunction with a recorder capability of 1000 cycle bandwidth can be used with any of the two above transducers (Strain Gage and L.V.D.T.)

10. SUMMARY AND RECOMMENDATIONS

It is concluded that under the contract a most useful tool has been developed to study and determine the energy required to initiate a reaction when various engineering materials come in contact with liquid oxygen, nitrogen tetroxide or other fuels and oxidizers needing additional evaluation.

Initial material testing indicated that when a reaction does take place, a considerable damage was inflicted in the test chamber. Our recommendation to correct this condition is to make the damaged parts expendible, namely by replacing the piston tip and insert sleeve in the wall of the test chamber housing as shown in Fig. 22.

A second recommendation is to provide a better sealing configuration and an easier manufacturing configuration by incorporating another Omniseal EX6802-2, also shown in Fig. 22, thereby eliminating any seal requirement located on the piston and making it essentially a one piece piston with a short removable tip.

It is also our recommendation that from our analysis of the routine testing program (Section 7 of this report) that the factorial design of the experiment as specified be considered in establishing future test programs with this tester.

The complete tester and temperature controller were shipped to Mr. C.F. Key, NASA, Huntsville, Alabama on May 24, 1966.

Robert Newcombe

R. Newcombe
Project Engineer

Approved:

W.B. Moen
Assistant Chief Engineer

7/21/66
sk/mb

Table 1. DYNAMIC PRESSURE RISE CALIBRATION

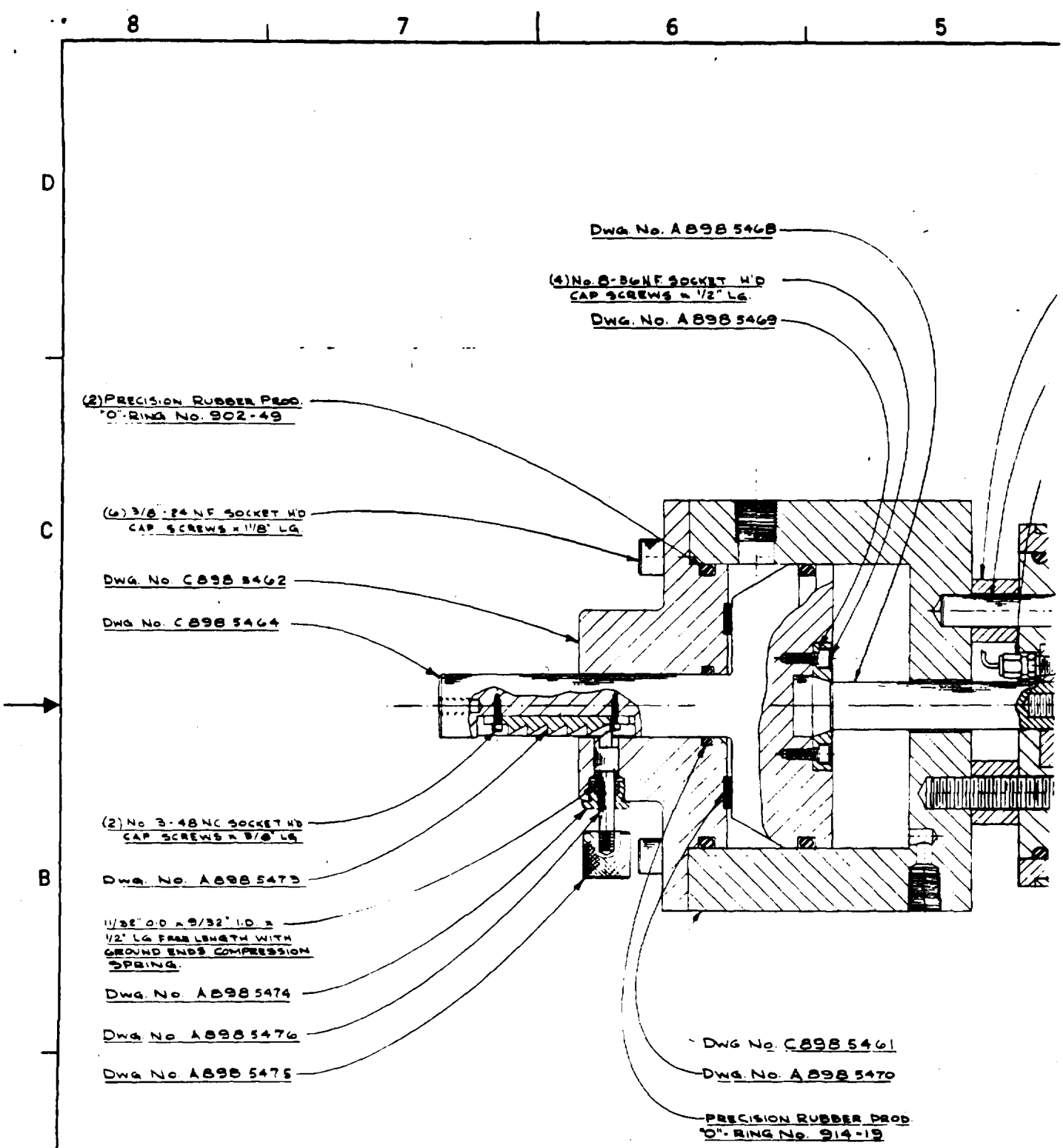
Pressure Applied (p.s.i.g.)	Rise Time (sec.)	Time Cycles	Calibrated Pressure Attained (p.s.i.g.)	Actual Pressure Attained (p.s.i.g.)	Rate of Pressure Rise Attained (p.s.i./sec.)
100	10.8×10^{-3}	0.65	3,400	3,230	299×10^3
200	14.19×10^{-3}	0.80	6,800	6,630	467×10^3
300	18.6×10^{-3}	1.12	10,200	10,200	548×10^3
400	18.75×10^{-3}	1.13	13,600	13,940	943×10^3
500	18.75×10^{-3}	1.13	17,000	17,000	906×10^3
600	19.6×10^{-3}	1.18	20,400	21,420	1093×10^3
700	18.3×10^{-3}	1.1	23,800	24,300	1328×10^3
800	20.75×10^{-3}	1.25	27,200	27,200	1310×10^3
900	19.9×10^{-3}	1.2	30,600	34,000	1708×10^3

Note: No orifice used between the high pressure surge tank and the driving chamber of the rig.

Table 2. n-PROPYL NITRATE DATA

Test No.	Drive Pressure (p.s.i.g.)	Drive Gas	Temp. (°F)	Bubble Size (ml.)	Sample Size (ml.)	Energy Input (kg-cm.)	Test Results
1	400	He (No Orifice)	65	0.8	1.0	12.1	Go
2	100	He (No Orifice)	65	0.8	1.0	3.68	No Go
3	150	He (No Orifice)	65	0.8	1.0	14.5	Go
4	125	He (No Orifice)	65	0.8	1.0	6.45	No Go
5	150	He (No Orifice)	65	0.6	1.0	7.47	Go
6	100	He (No Orifice)	65	0.6	1.0	--*	Go
7	50	He (No Orifice)	65	0.6	1.0	3.27	No Go

* Potentiometer damaged.


8
7
6
5
D
C
B
A
Dwg. No. A898 5468
(4) No. 8-36 N.F. Socket H'D Cap Screws x 1/2" Lg.
Dwg. No. A898 5469
(2) Precision Rubber Prod. "O"-Ring No. 902-49
(6) 3/8-24 N.F. Socket H'D Cap Screws x 1 1/8" Lg.
Dwg. No. C898 5462
Dwg. No. C898 5464
(2) No. 3-48 N.C. Socket H'D Cap Screws x 3/8" Lg.
Dwg. No. A898 5473
11/32" O.D. x 9/32" I.D. x 1/2" Lg. Free Length with Ground Ends Compression Spring.
Dwg. No. A898 5474
Dwg. No. A898 5476
Dwg. No. A898 5475
Dwg. No. C898 5461
Dwg. No. A898 5470
Precision Rubber Prod. "O"-Ring No. 914-19

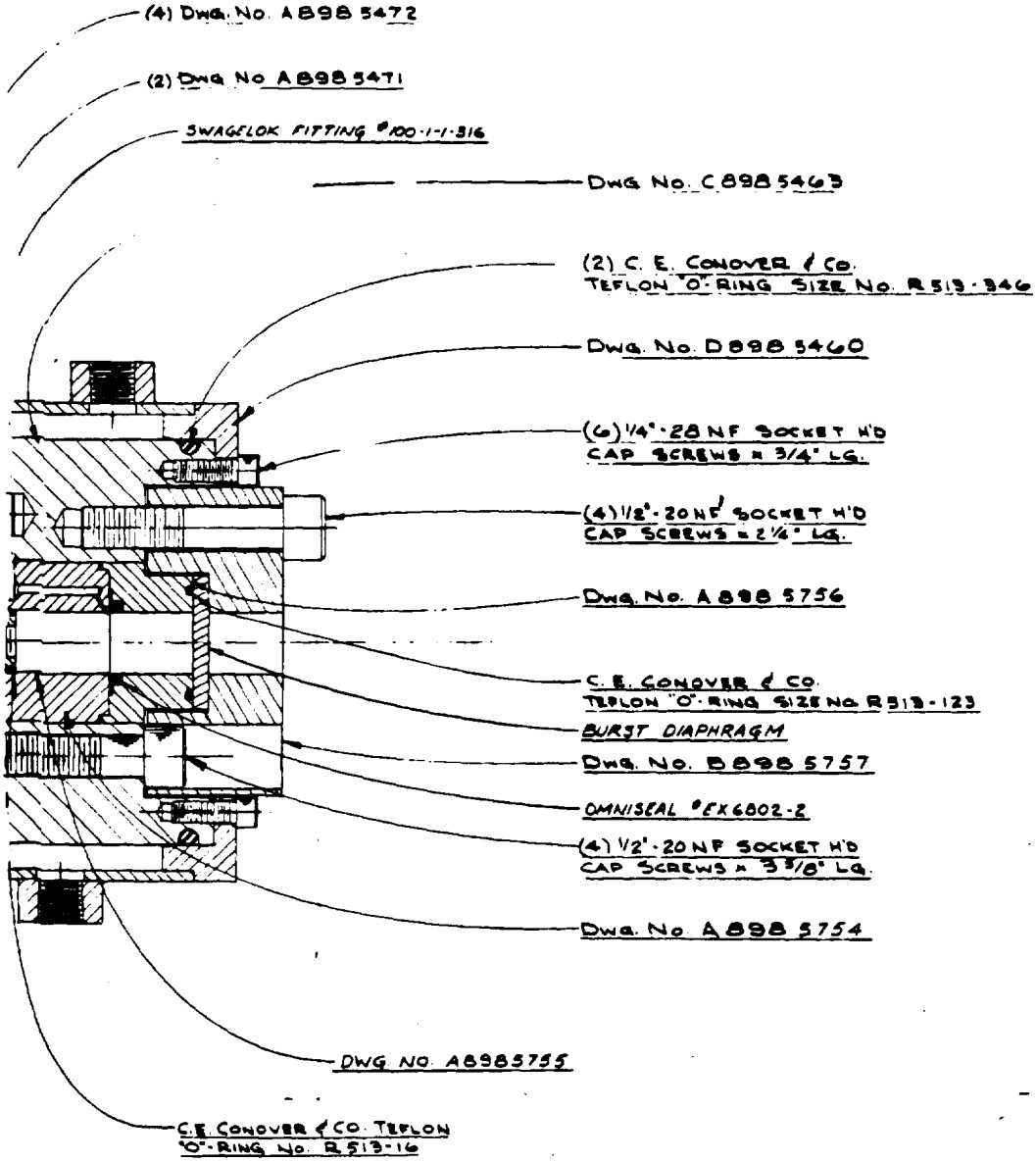


Fig. 1.

TOLERANCES DEC ± .005 FRACT.	TITLE ADIABATIC COMPRESSION TESTER (MAIN ASSEMBLY)		
REVISIONS	ANGLES	SCALE FULL	SUPERSEDES NO.
C. R. M.			SUPERSEDED BY NO.
AIRCO AIR REDUCTION COMPANY, INC.		D.E. NO.	DWG. LIST
		SIZE D	DRAWING NO. 898 5454
		SHEET NO.	OF SHEETS
		REV. 2	

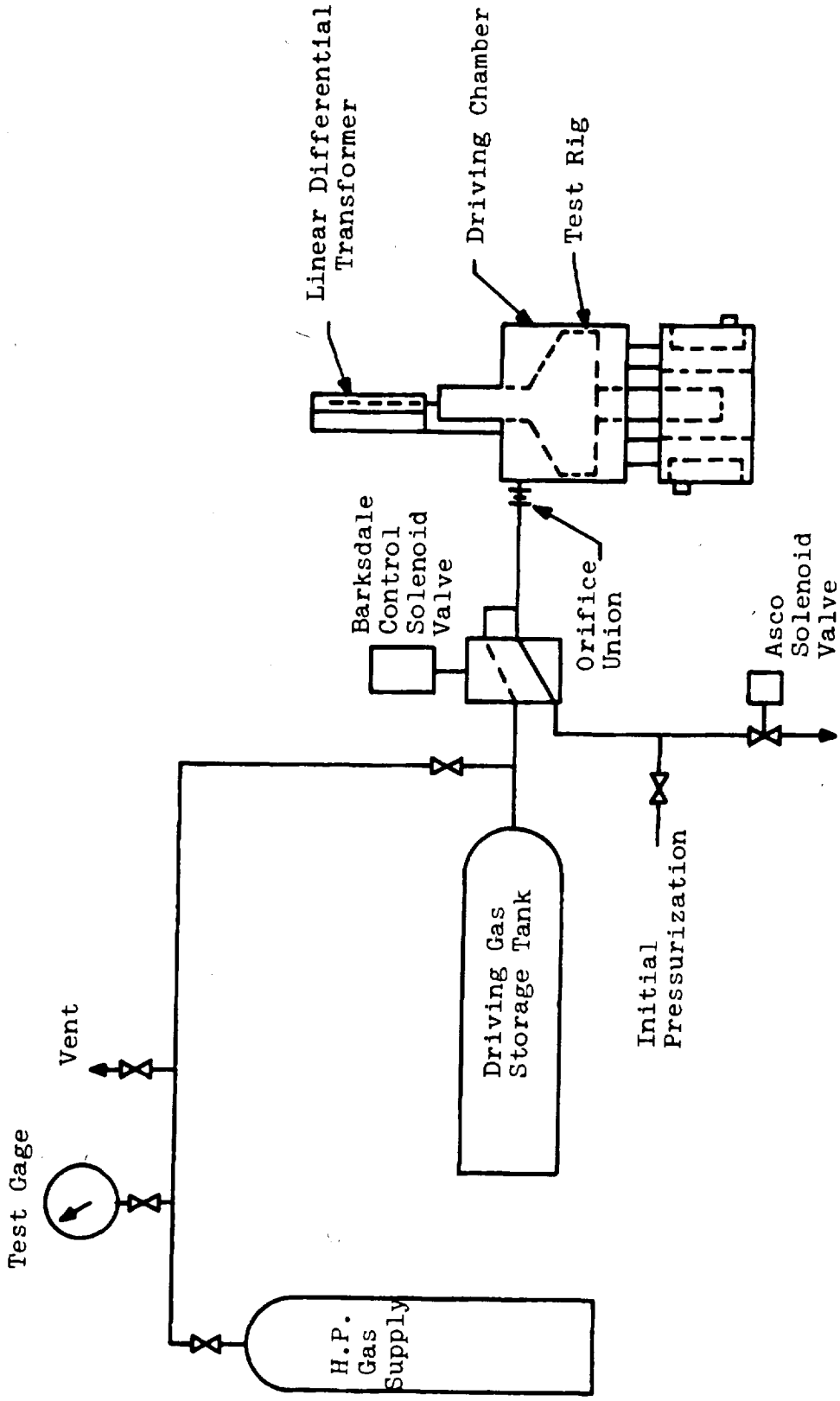
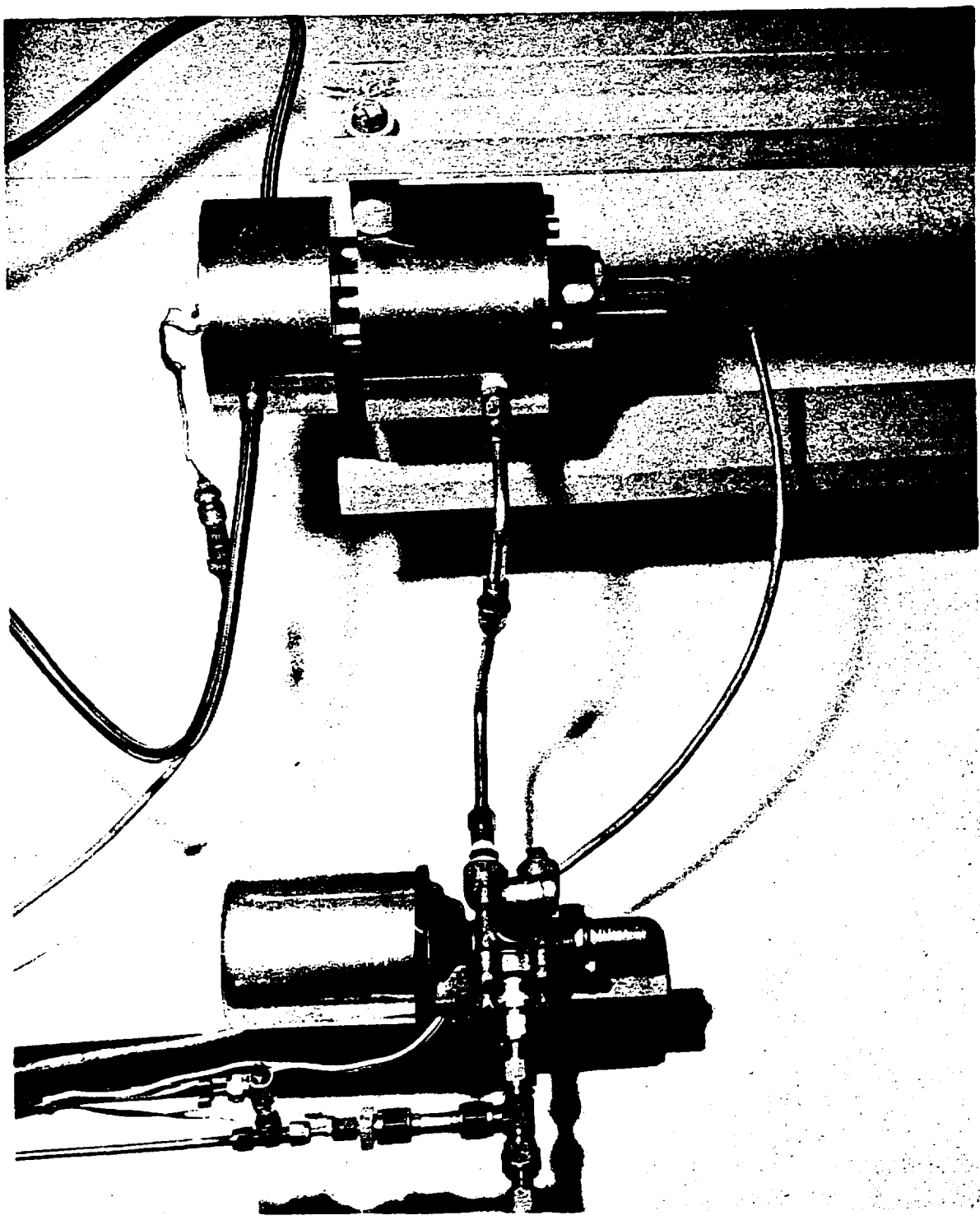


Fig. 2. ADIABATIC COMPRESSION TEST RIG PNEUMATIC SCHEMATIC DIAGRAM



15622

Fig. 3. ADIABATIC COMPRESSION TEST RIG PHEUMATIC SYSTEM

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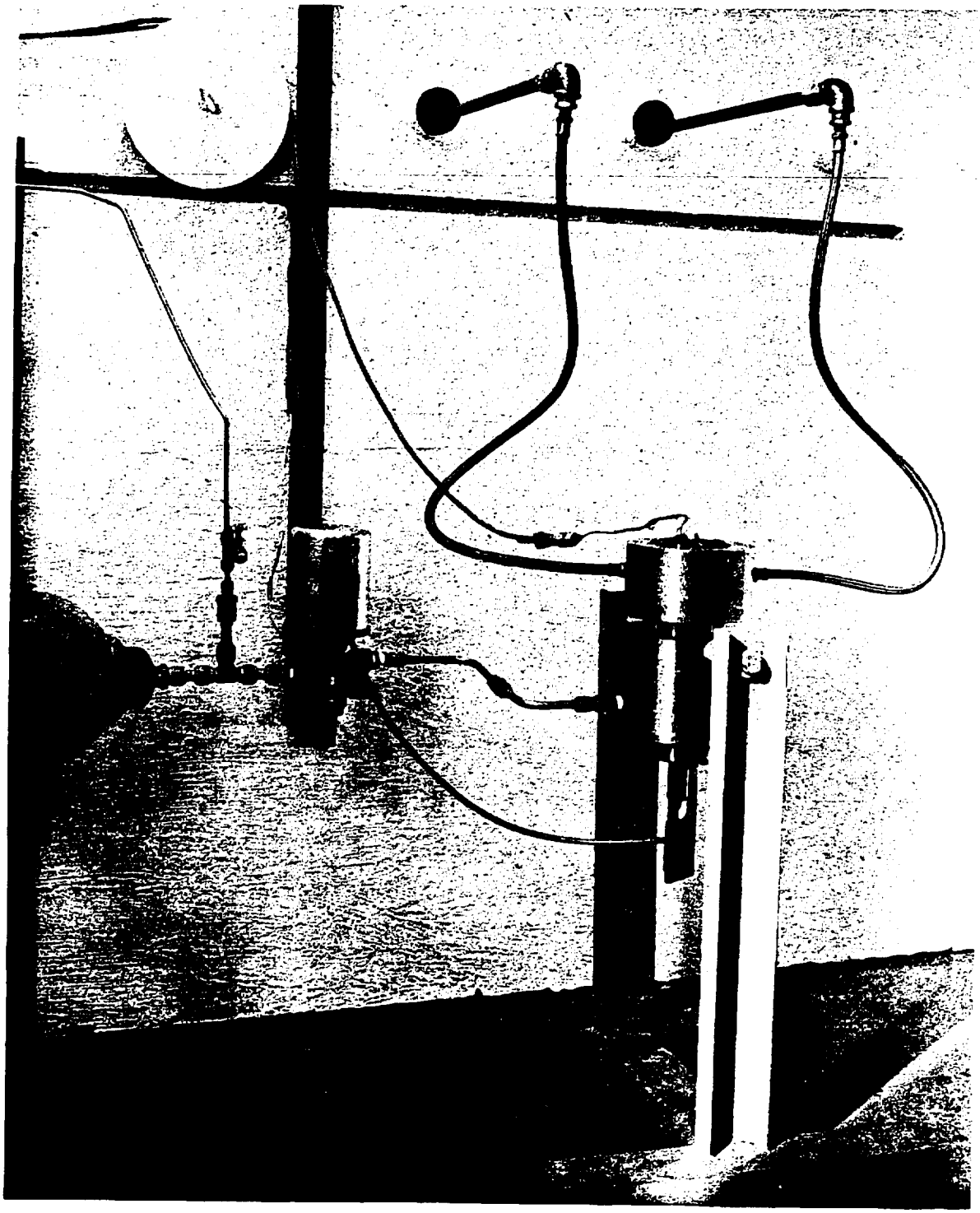


15621

Fig. 4. ADIABATIC COMPRESSION TEST RIG PHEUMATIC SYSTEM

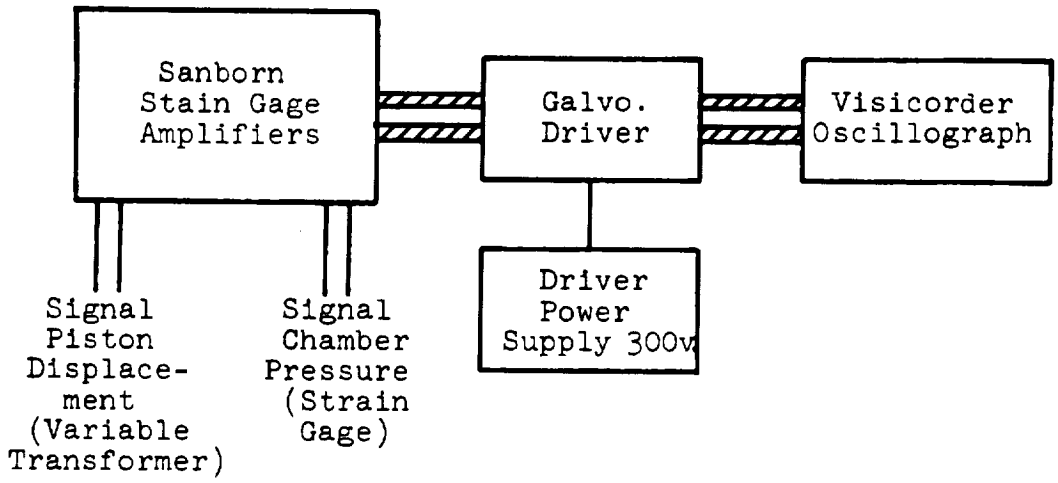
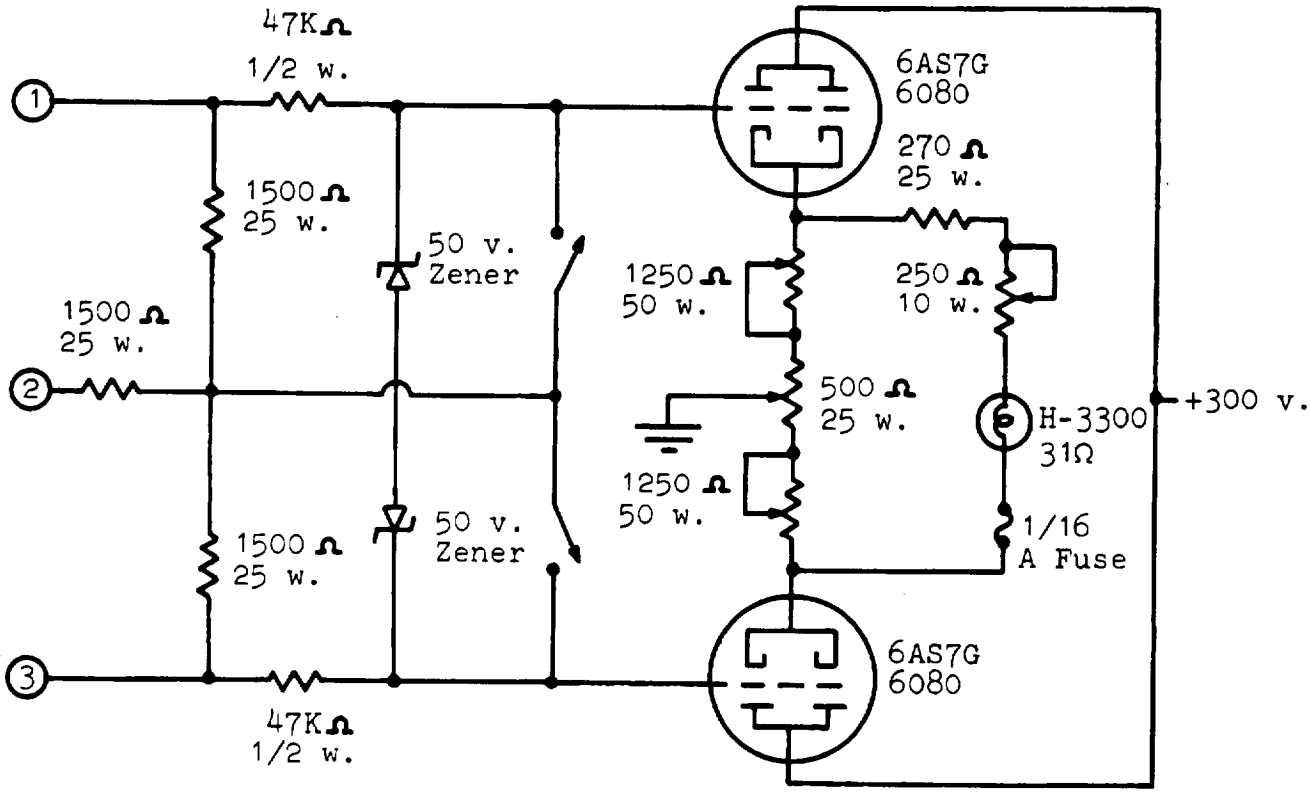


Fig. 5a. INSTRUMENTATION SCHEMATIC DIAGRAM



This unit duplicated for each galvonometer

Fig. 5b. GALVONOMETER DRIVER SCHEMATIC DIAGRAM

Fig. 5. INSTRUMENTATION SCHEMATIC

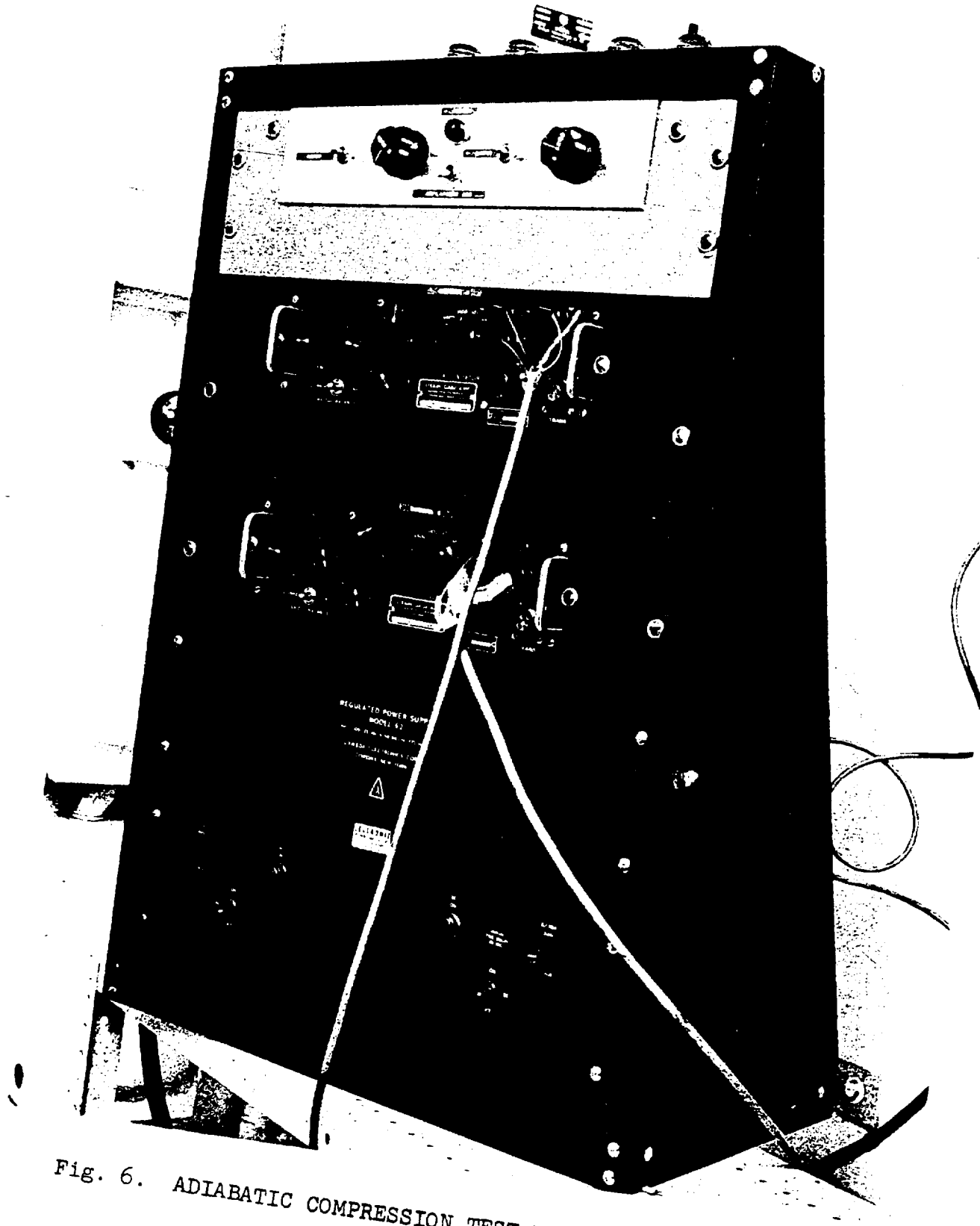
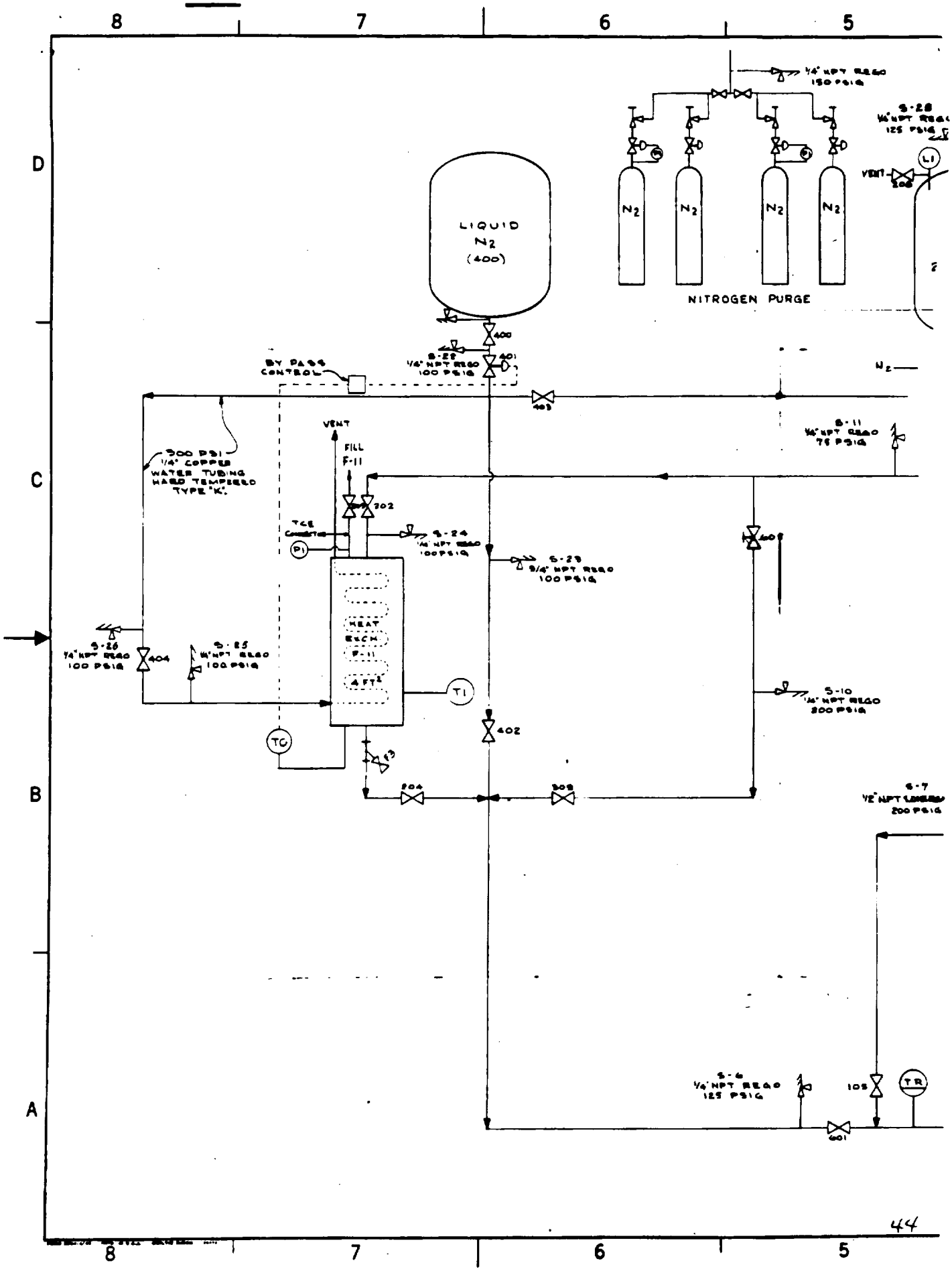


Fig. 6. ADIABATIC COMPRESSION TEST RIG INSTRUMENT AMPLIFIERS 15623


8
7
6
5
D
C
B
A
LIQUID
N2
(400)
N2
N2
N2
N2
NITROGEN PURGE
1/4" NPT REGO
150 PSIG
S-28
1/4" NPT REGO
125 PSIG
LI
VENT
200
400
401
S-22
1/4" NPT REGO
100 PSIG
BY PASS
CONTROL
403
N2
S-11
1/4" NPT REGO
75 PSIG
300 PSI
1/4" COPPER
WATER TUBING
HARD TEMPERED
TYPE "K"
VENT
FILL
F-11
302
TCE
CONNECTION
P1
S-24
1/4" NPT REGO
100 PSIG
S-23
1/4" NPT REGO
100 PSIG
400
HEAT
EXCH.
F-11
4 FT²
T1
S-26
1/4" NPT REGO
100 PSIG
404
S-25
1/4" NPT REGO
100 PSIG
TC
402
S-10
1/4" NPT REGO
200 PSIG
S-7
1/4" NPT REGO
200 PSIG
S-6
1/4" NPT REGO
125 PSIG
105
TR
401

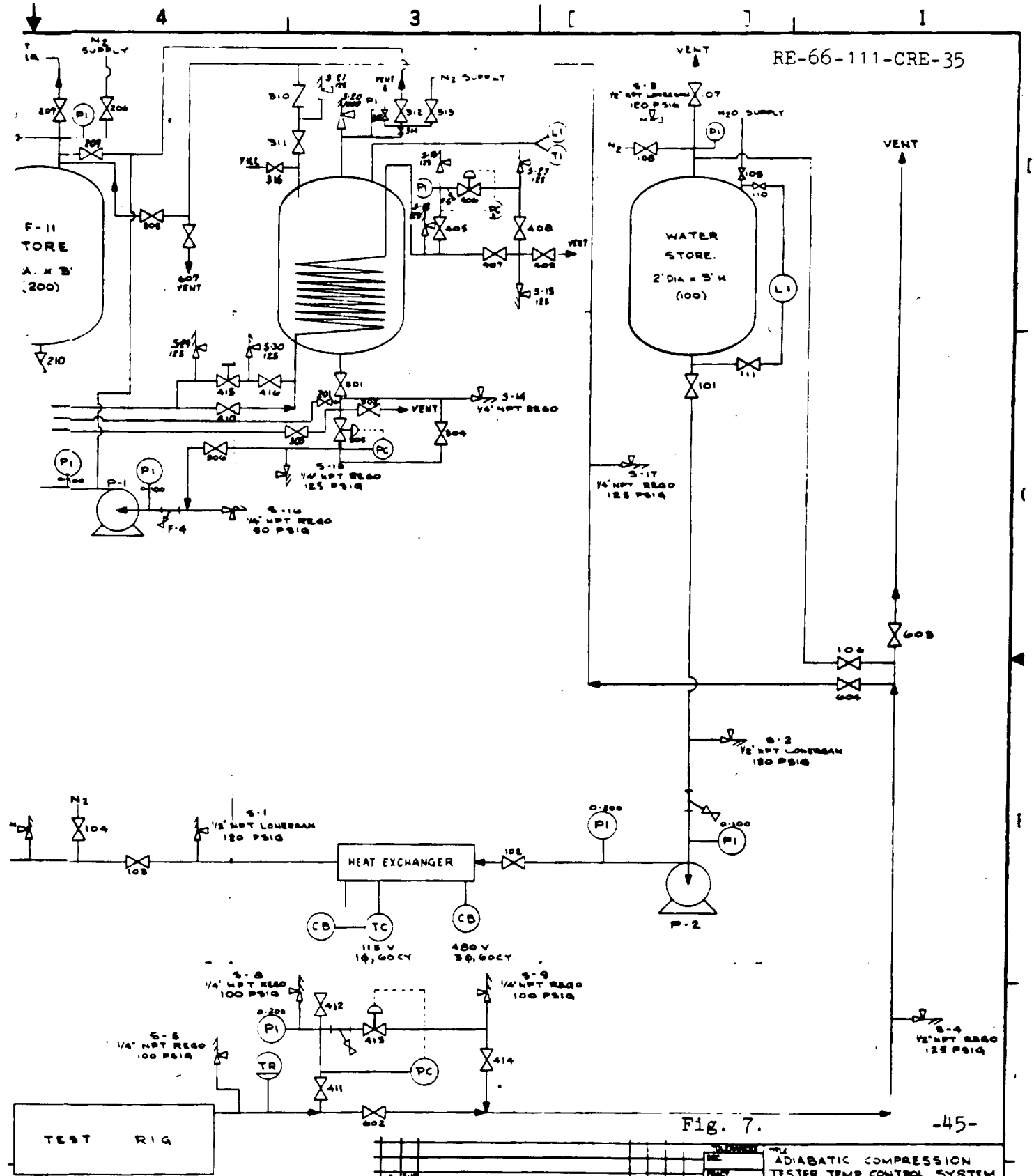

F-11
TORE
A. & B'
(200)
WATER
STORE.
2' DIA x 3' H
(100)
VENT
HEAT EXCHANGER
TEST RIG
P-1
P-2
115 V
1φ, 60 CY
480 V
3φ, 60 CY
ADIABATIC COMPRESSION
TESTER TEMP. CONTROL SYSTEM
AIR REDUCTION
COMPANY, INC.
898 5456

Fig. 7.

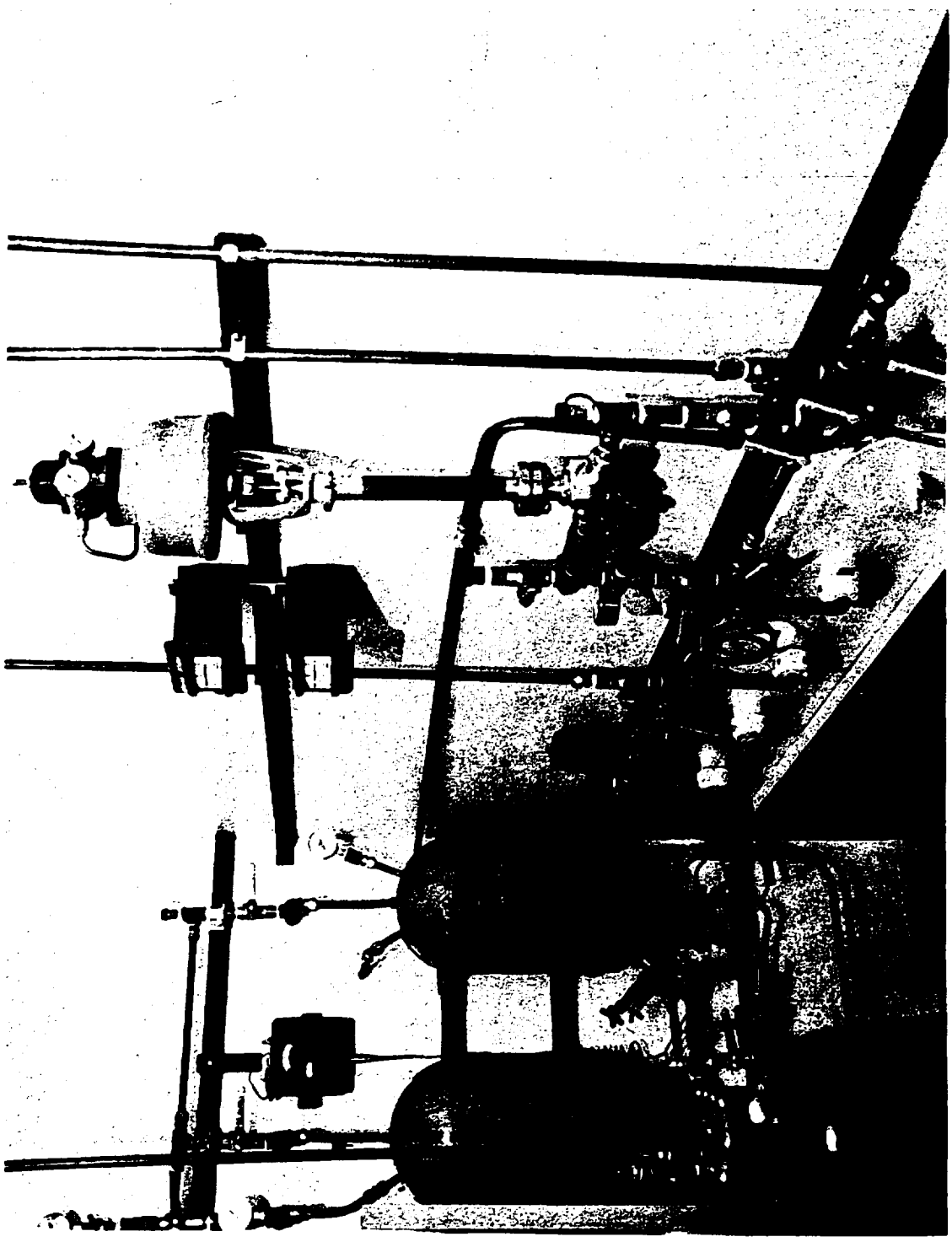


Fig. 8. TEMPERATURE INDICATORS and PRESSURE CONTROLLER

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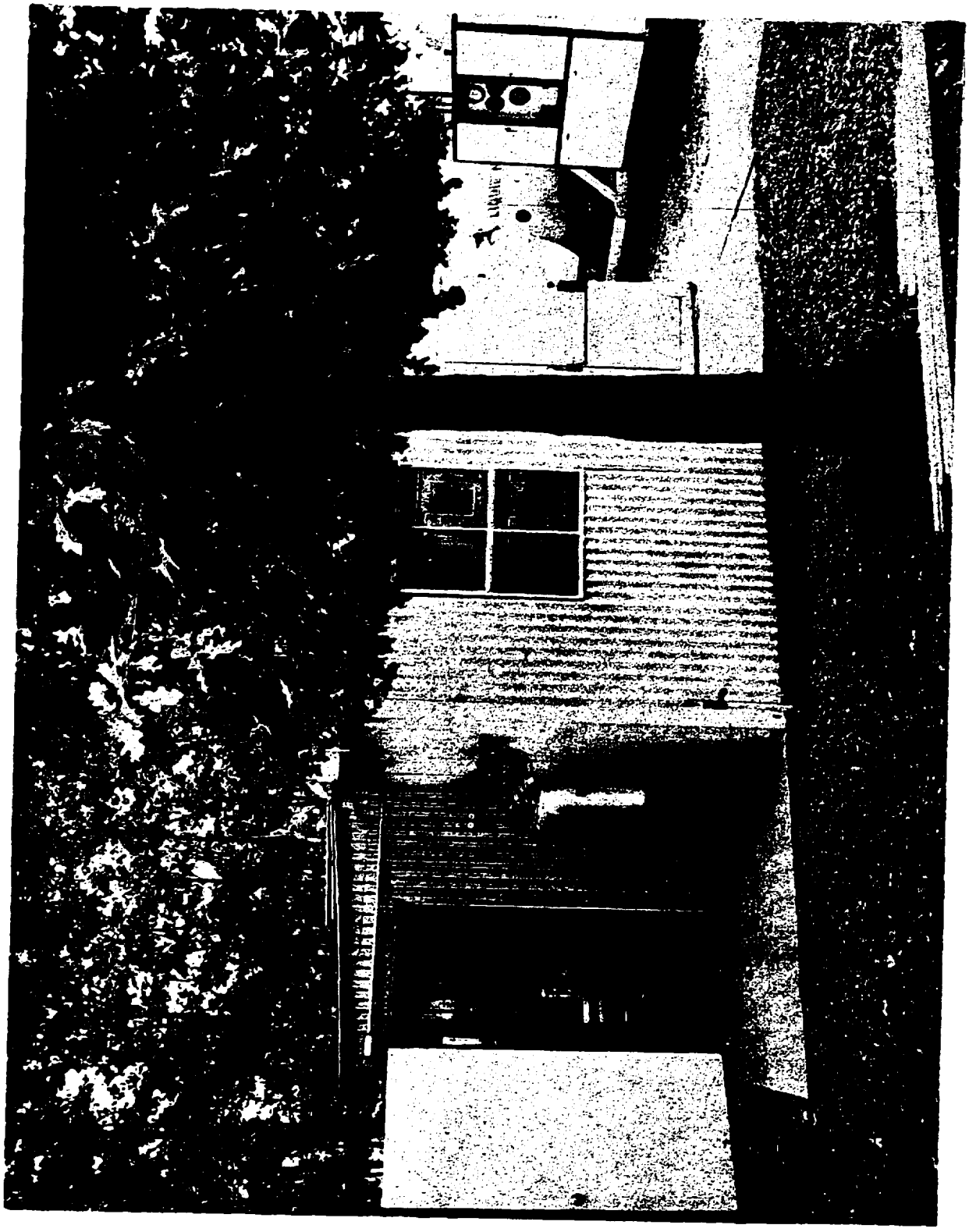

Fig. 9. AIRCO TEST FACILITY INSTALLATION

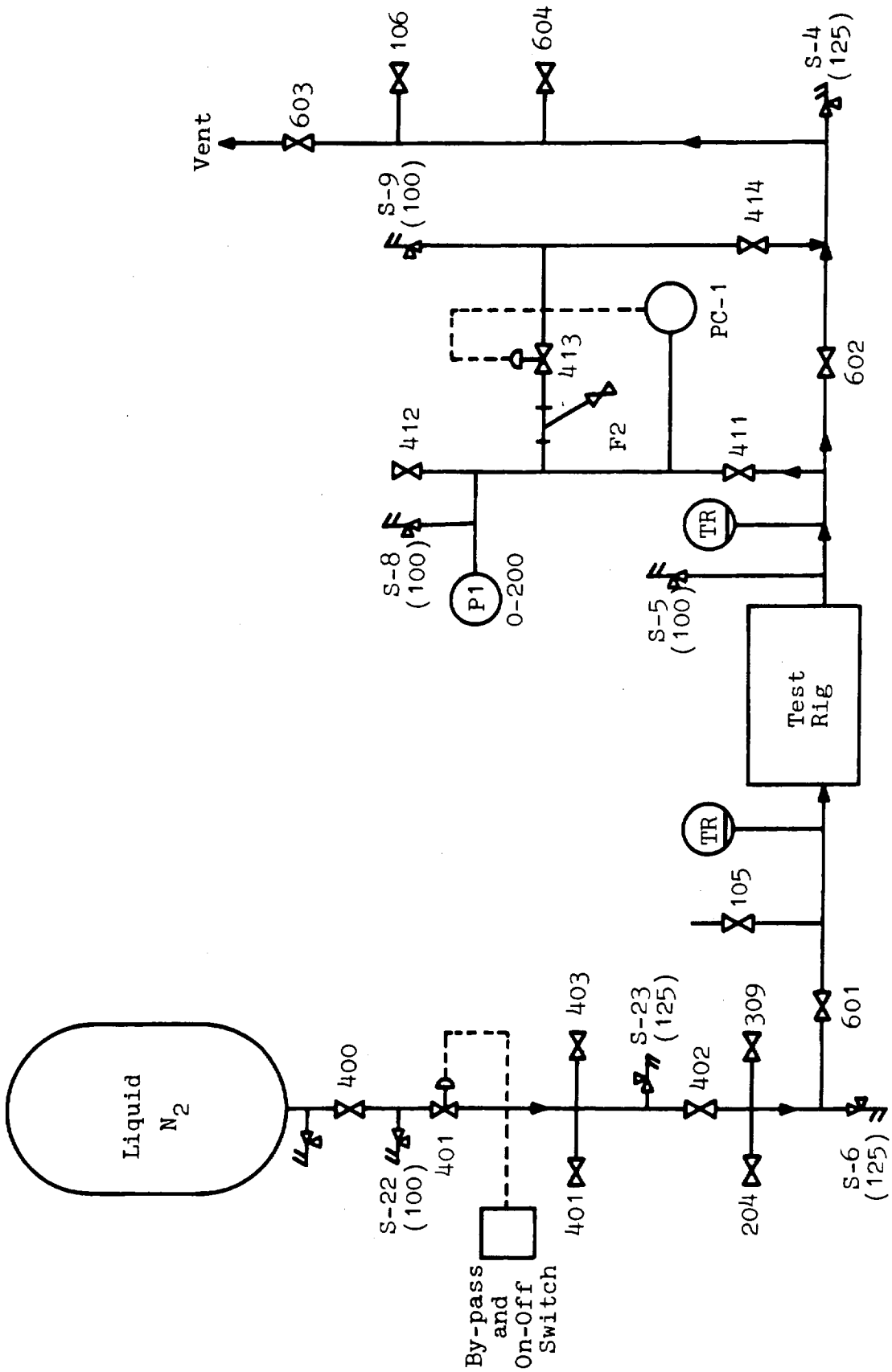

Fig. 10. TEMPERATURE CONTROL SYSTEM: LIQUID NITROGEN FLOW DIAGRAM

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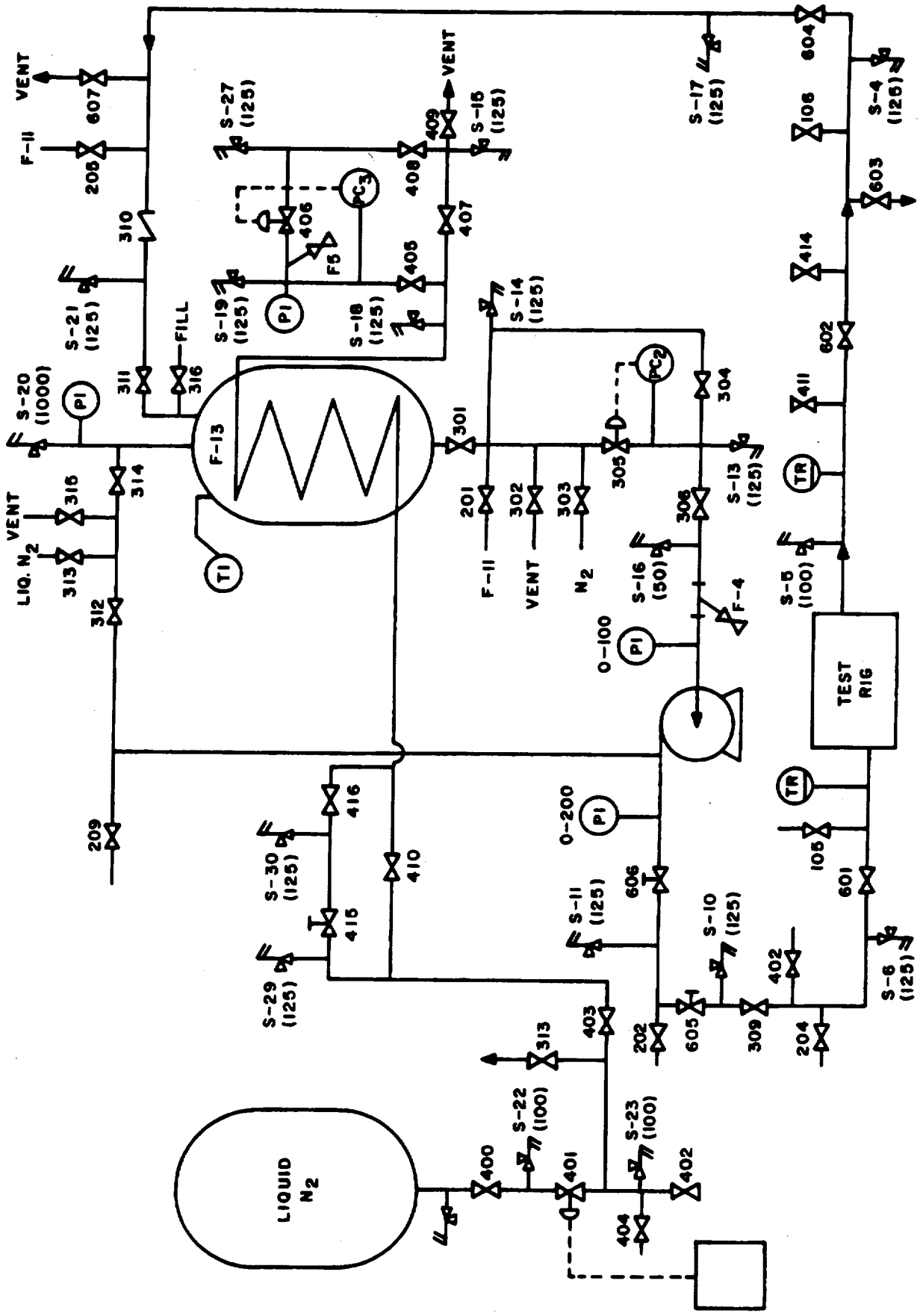

Fig. 11. TEMPERATURE CONTROL SYSTEM: FREON-13 FLOW DIAGRAM

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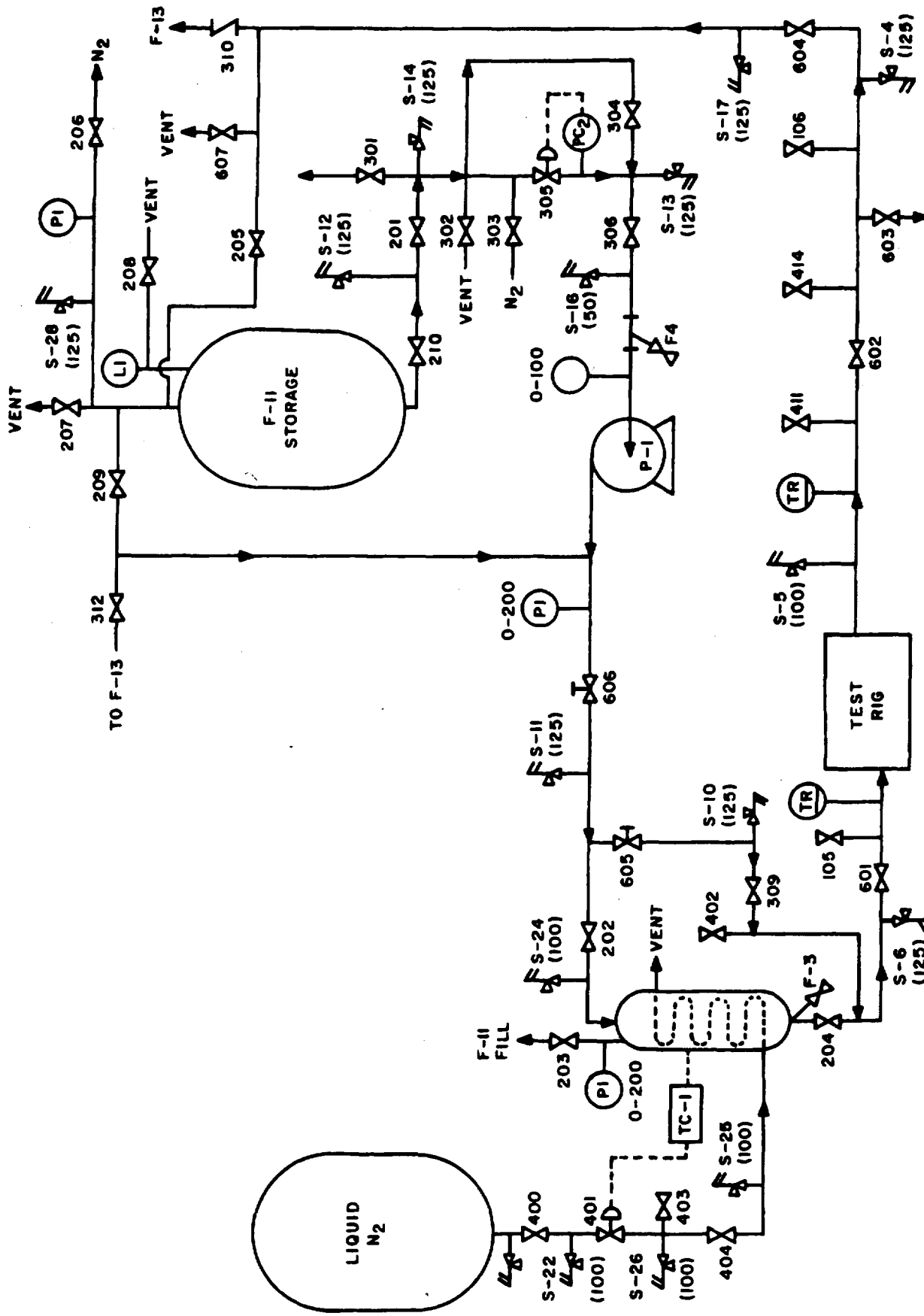

Fig. 12. TEMPERATURE CONTROL SYSTEM: FREON-11 FLOW DIAGRAM

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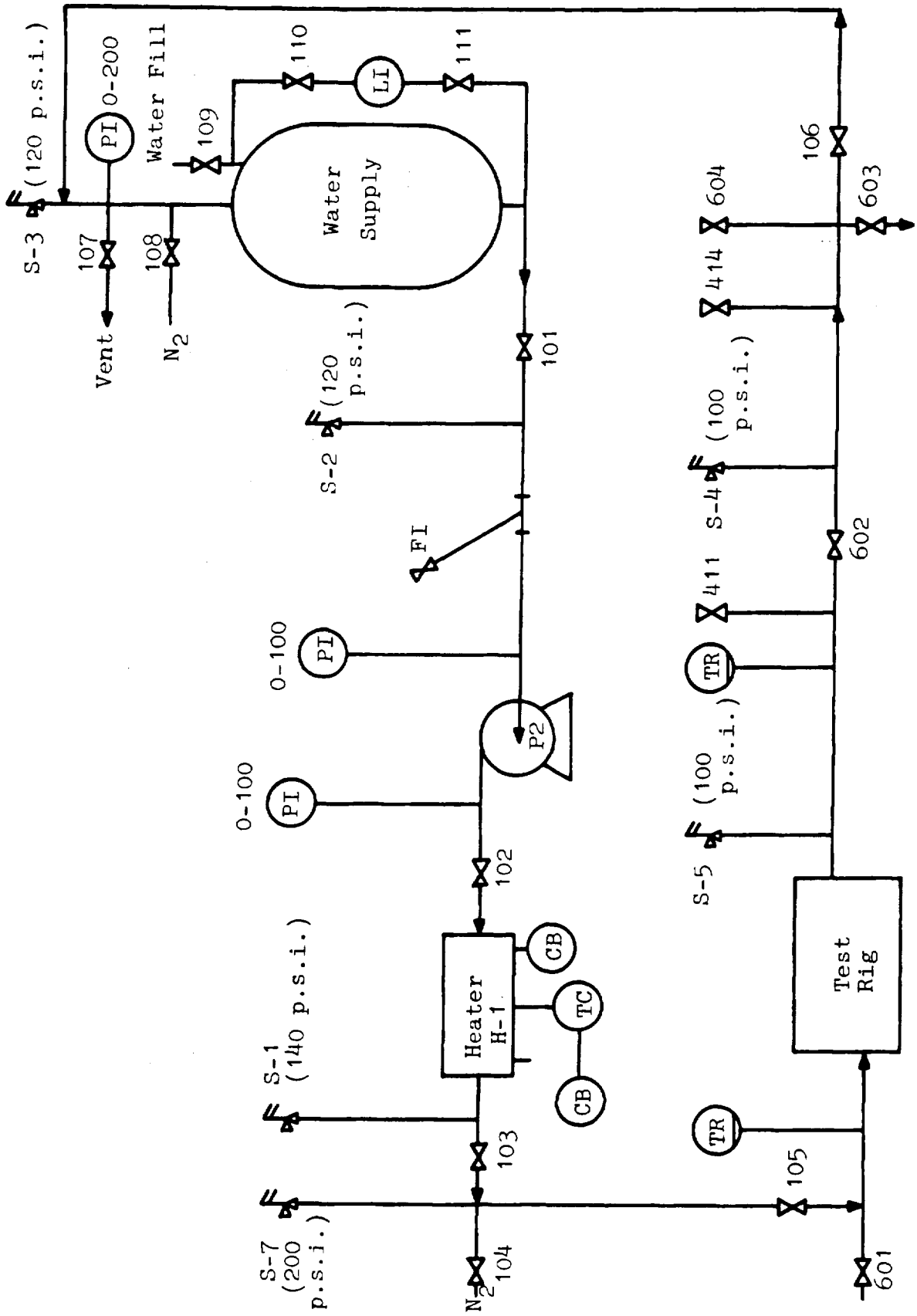

Fig. 13. TEMPERATURE CONTROL SYSTEM: WATER FLOW DIAGRAM

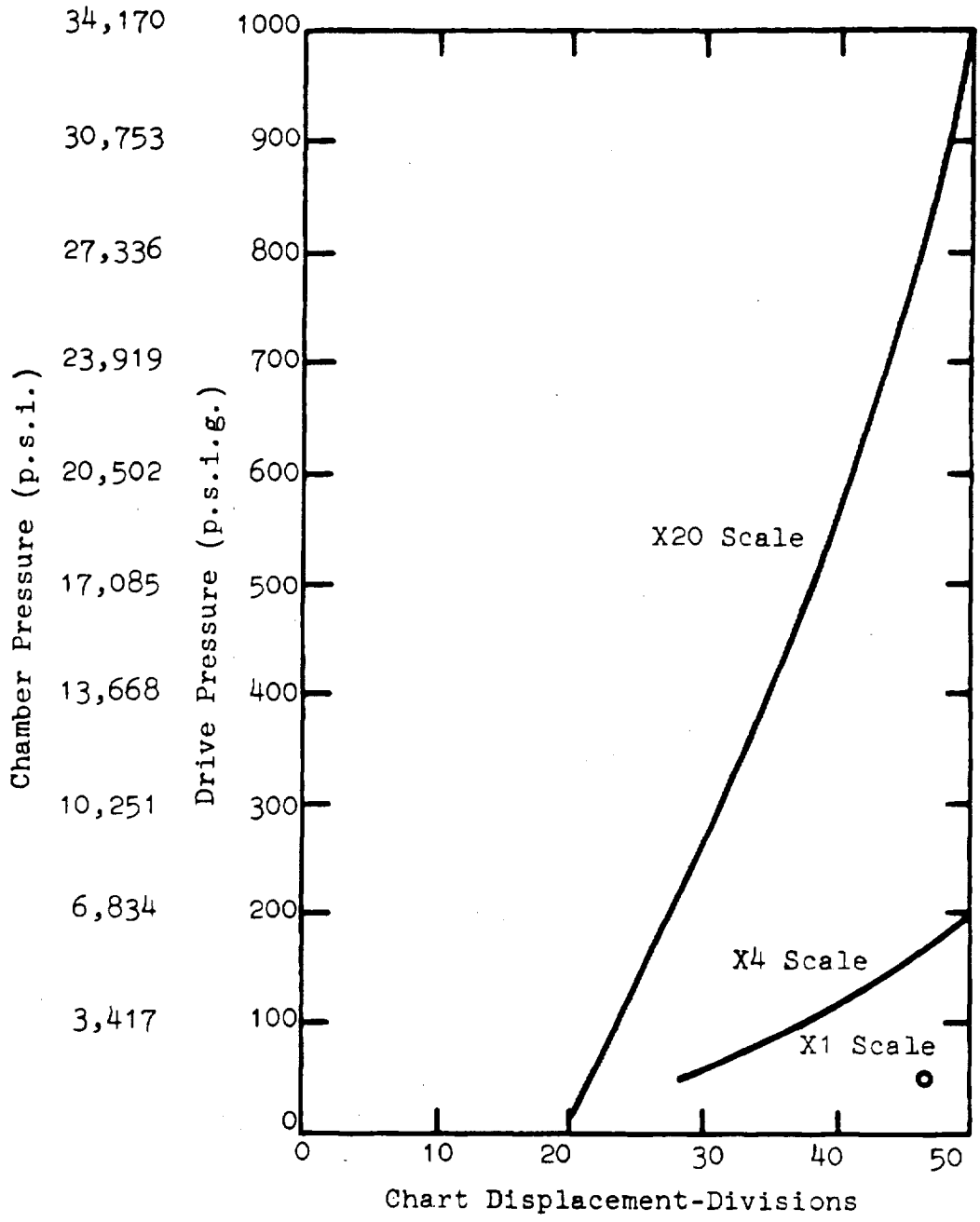

Fig. 14. CALIBRATION: PRESSURE vs. CHART READING

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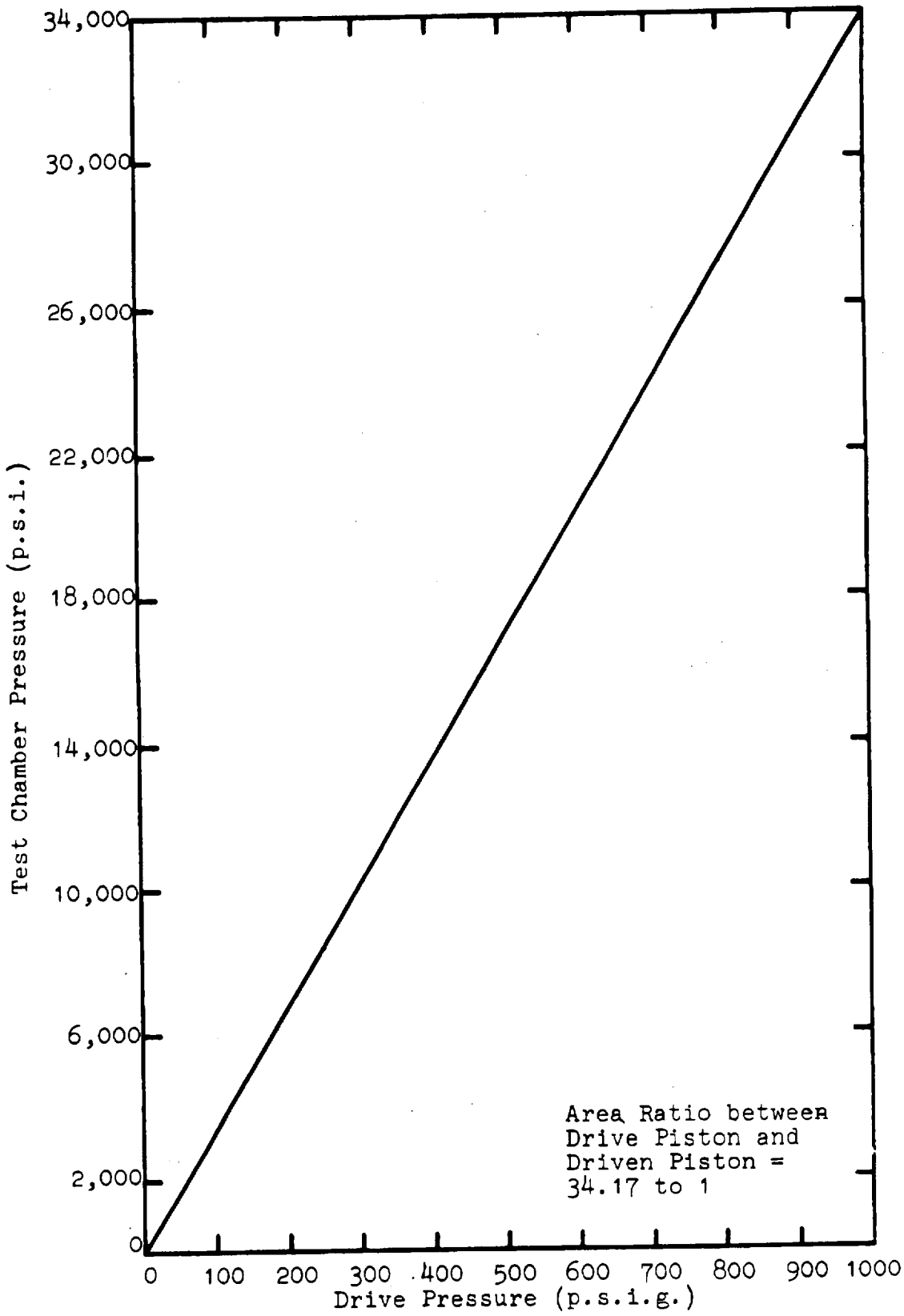

Fig. 15. CALIBRATION: PISTON DRIVE PRESSURE (p.s.i.g.) vs. TEST CHAMBER PRESSURE (p.s.i.)

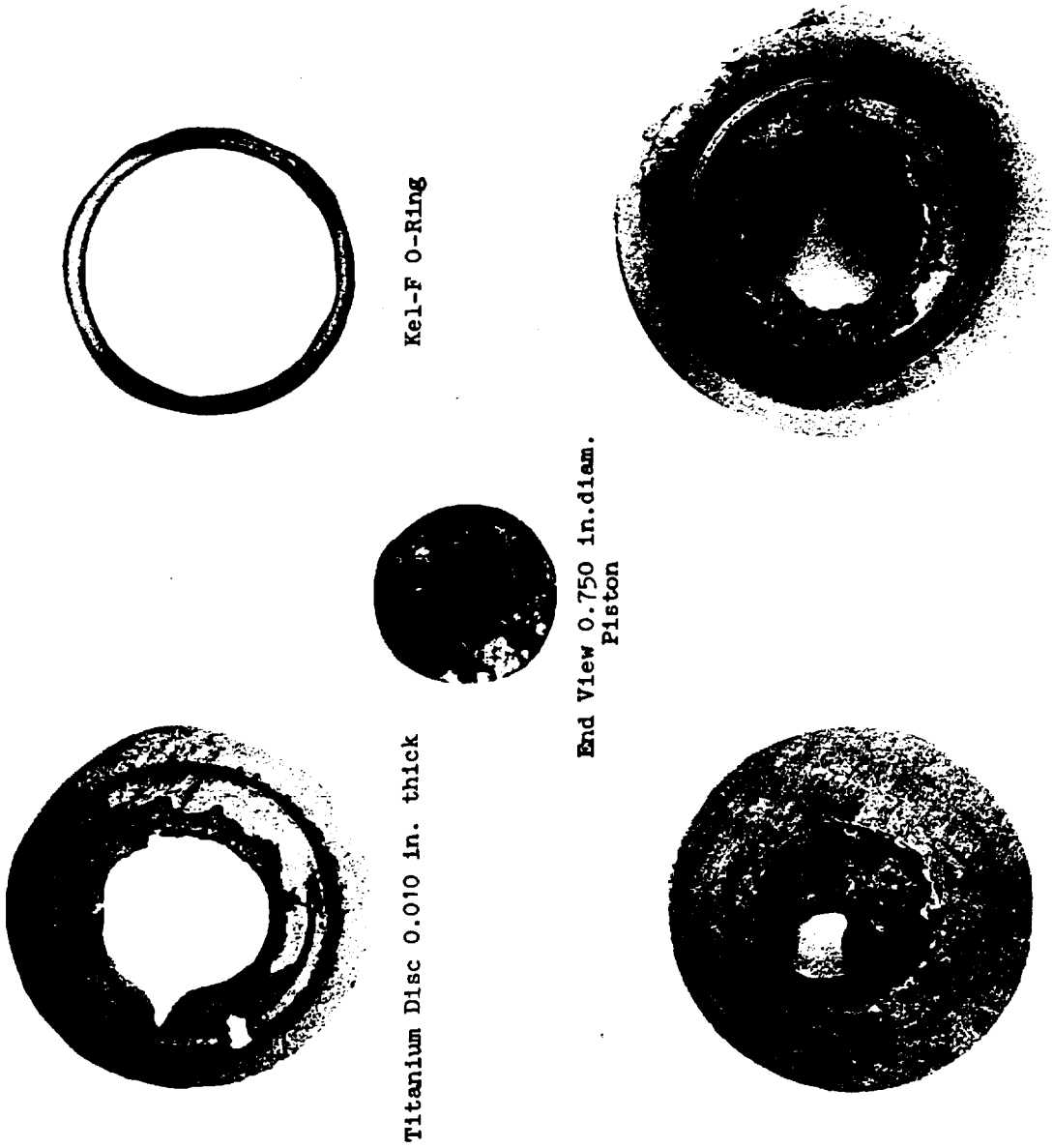

Fig. 16. RESULTS of a TITANIUM-OXYGEN REACTION

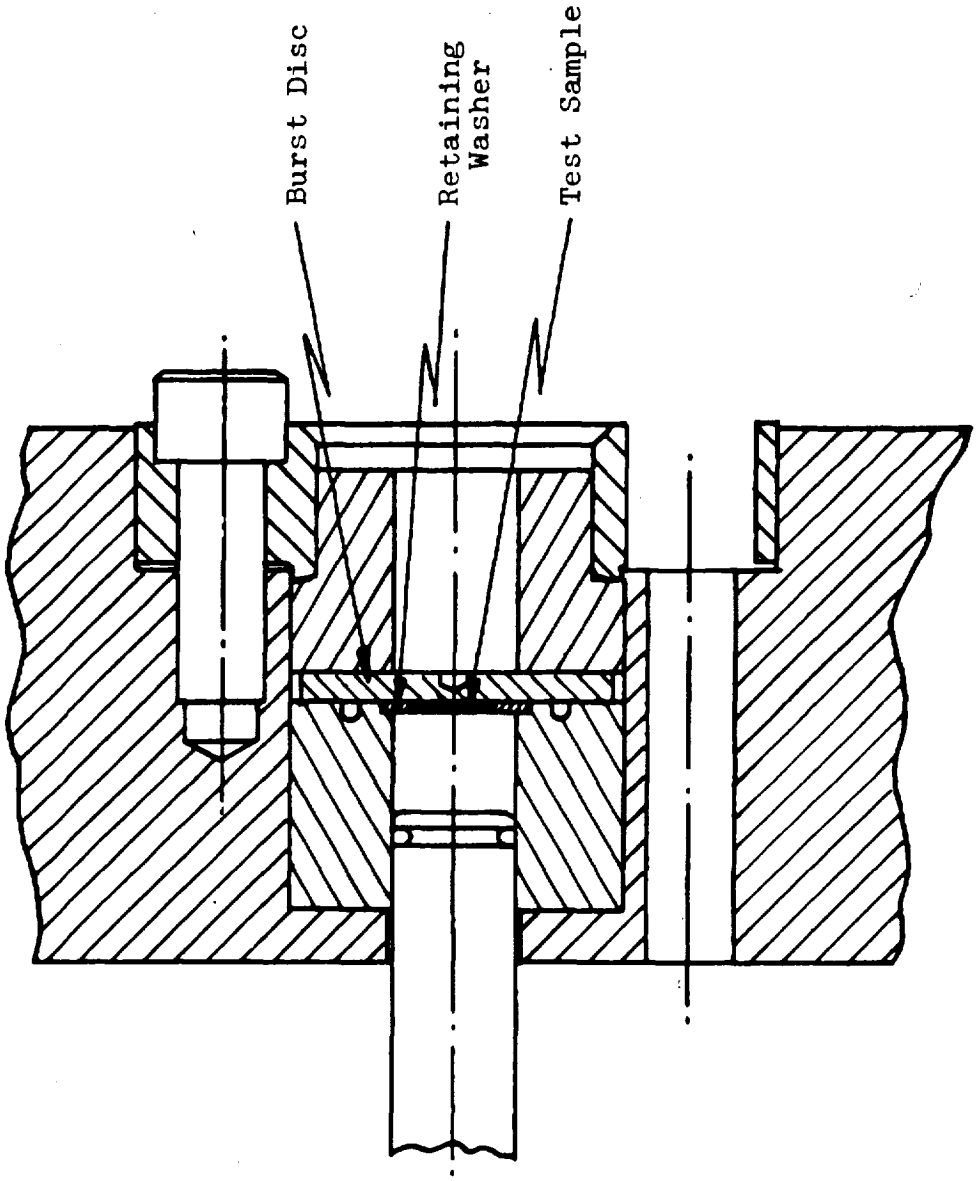

Fig. 17. MODIFIED SAMPLE RETAINER

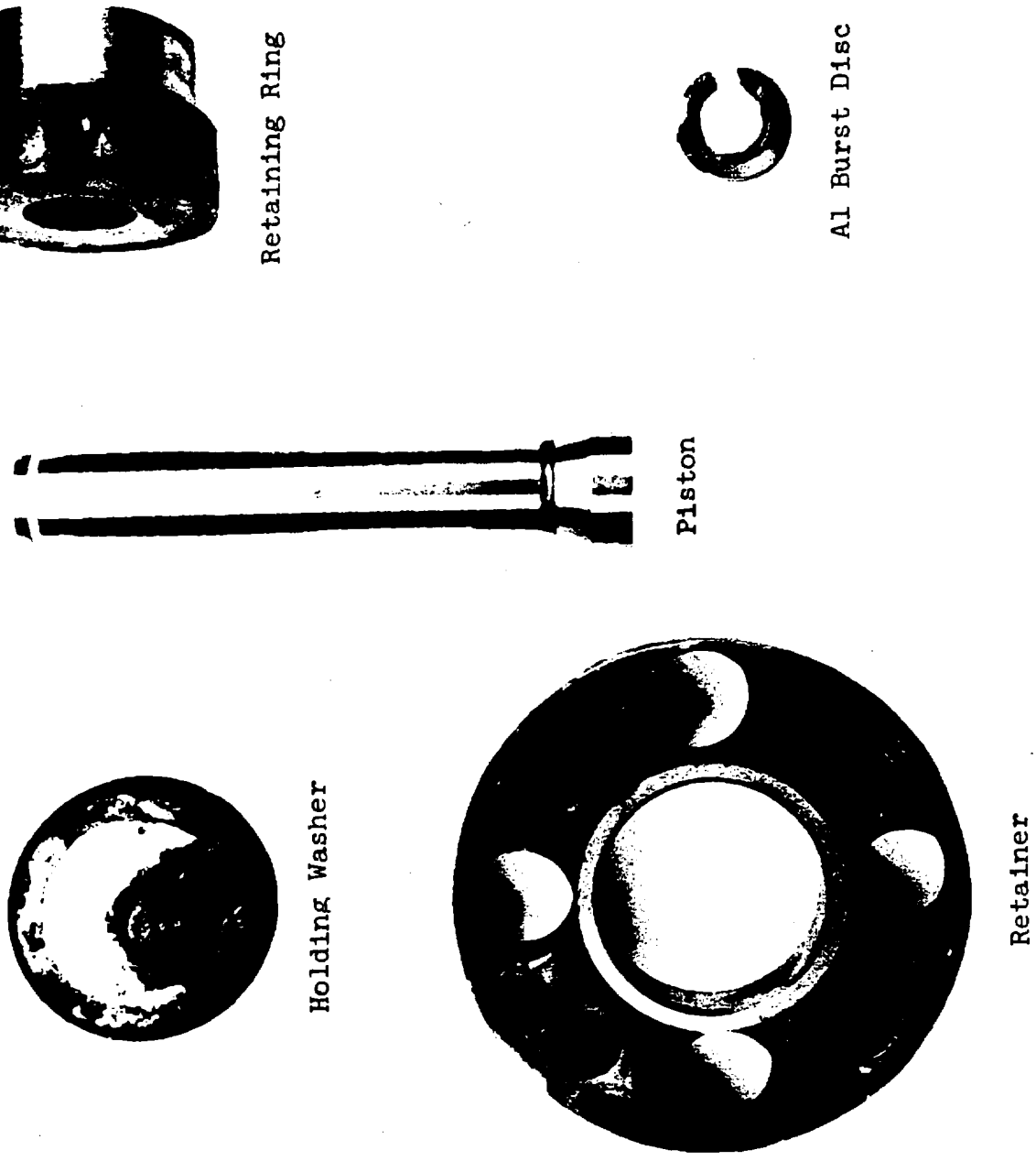

Fig. 18. DAMAGE to TESTER DUE to a POSITIVE REACTION

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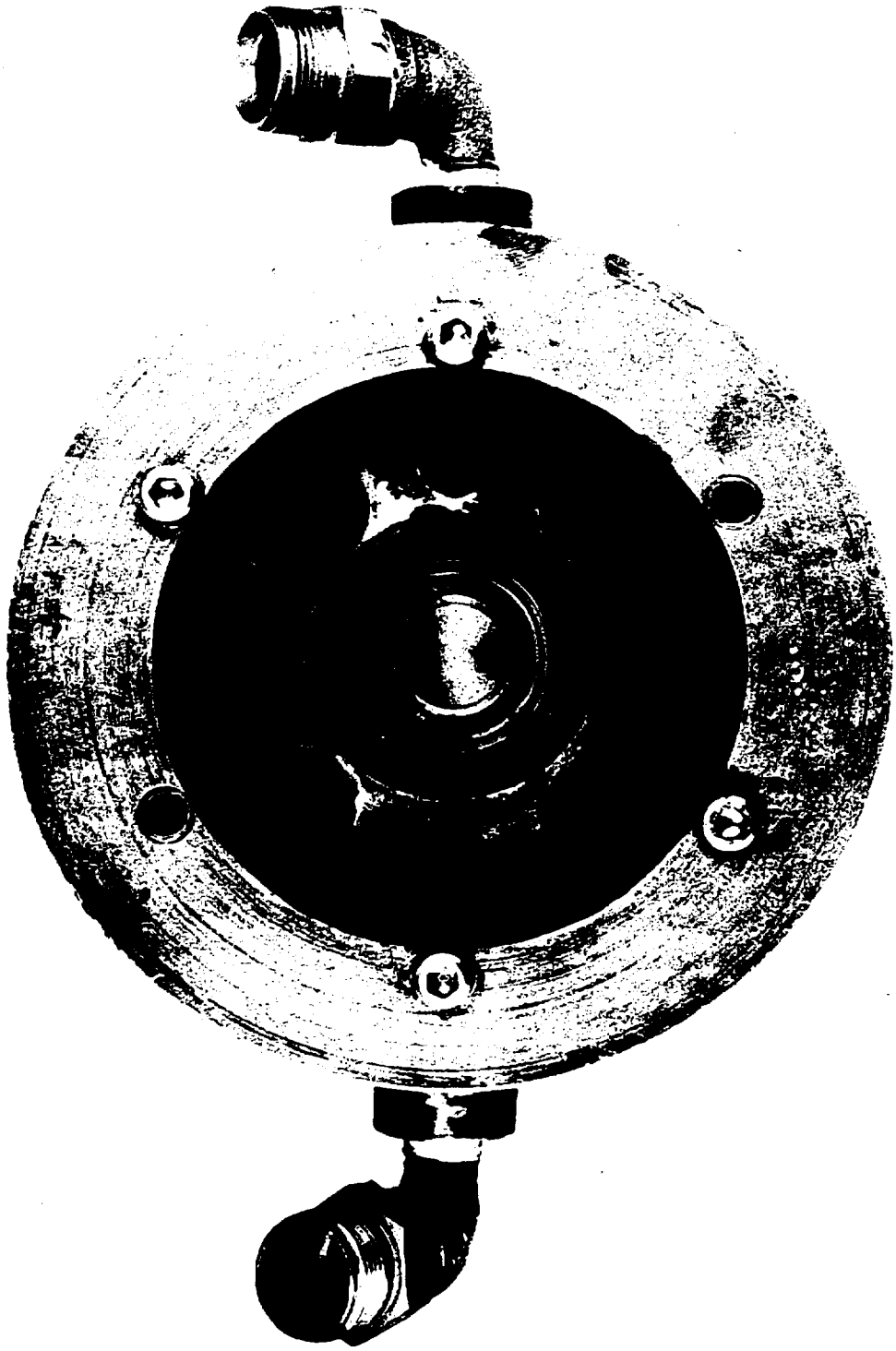
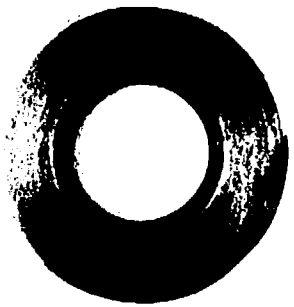


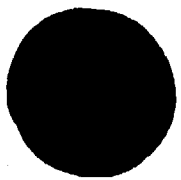
15714

Fig. 19. DAMAGE TO TESTER HOUSING DUE TO A POSITIVE REACTION

Al Burst Disc



Holding
Washer



Titanium
Sample
0.010 in. thick

15725

Fig. 20. DAMAGE to TESTER DUE to a POSITIVE REACTION:
TITANIUM and OXYGEN

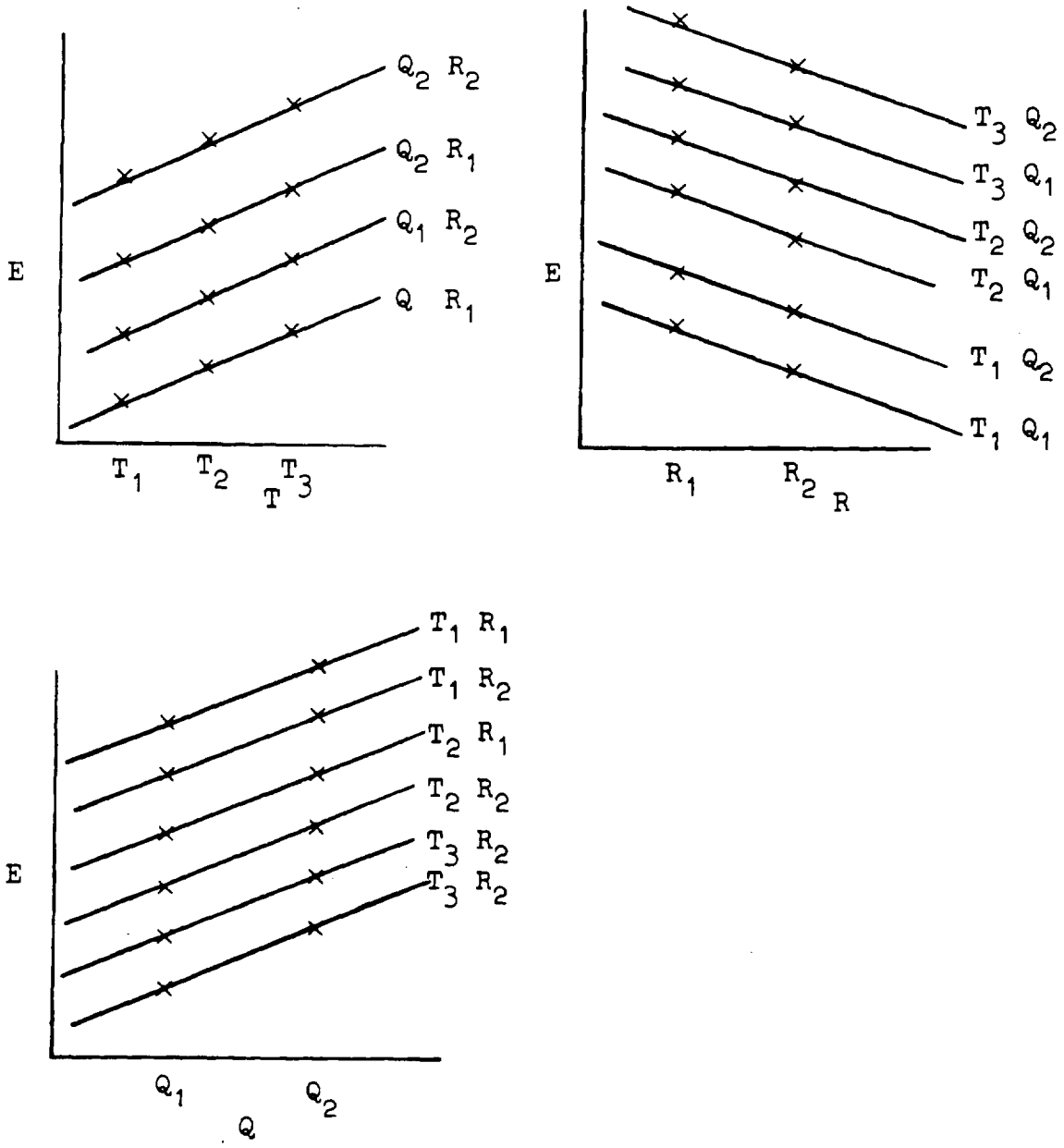


Fig. 21. TYPICAL DATA PLOTS of the FACTORIAL DESIGN EXPERIMENT

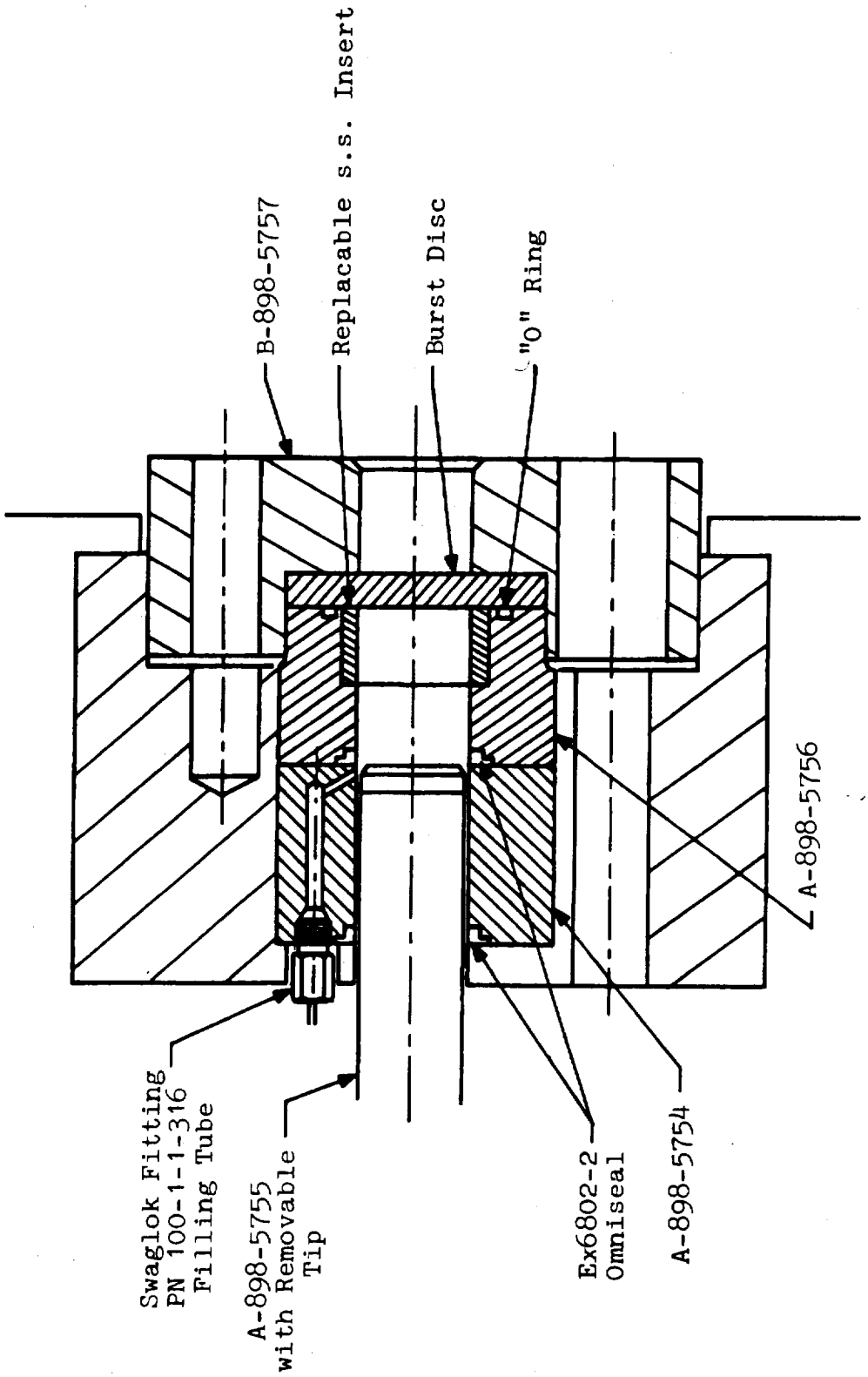


Fig. 22. RECOMMENDED FUTURE REWORK DESIGN of TEST CHAMBER

APPENDIX A. Literature Survey

1. UNCLASSIFIED LITERATURE SOURCES

At the start of the program a literature search was made in order to obtain information and background on adiabatic compression testing methods. Although some of the earlier testing procedures are applicable to solid explosives testing, the investigation was specifically limited to testing methods and procedures employed in testing liquid mono-propellants. Also, references are included that refer to the theoretical mechanisms involved in the initiation and growth of explosions.

The sources consulted in this search were as follows:

Battelle Technical Review	1958 - 1963
Chemical Abstracts	1946 - 1964
Dissertation Abstracts	1959 - 1964
Engineering Index	1958 - 1964
Index of NASA Publications	1958 - 1962
Science Abstracts/Physics Abstracts	1958 - 1964
Scientific & Technical Aerospace Reports (STAR)	1958 - 1965
Technical Abstract Bulletin	1959 - 1965

In connection with the use of the Technical Abstract Bulletin, the Defense Documentation Center, New York Field Office, provided access to the documents located by use of the bulletin, which were of an unclassified nature. Since the majority of references on adiabatic testing were classified either by the originator or the government, it was impossible to thoroughly abstract the documents so listed since this contract is unclassified. However, a list of related classified literature has been compiled and is included. There is also a number of unclassified reports of which copies have been requested but have not been received at this time. Abstracts of this literature will be included in later reports as they become available, and if applicable.

The following is a presentation of the results of the literature search in the form of a bibliography and abstracts of pertinent subject matter contained in the documents.

(1a) BOWDEN, F.P., MULCAHY, M.F.R., VINES, R.G. and YOFFE, A.
The detonation of liquid explosives by gentle impact: The effect of minute gas spaces.
Proc.Roy.Soc.(London) A188: 291-311 (1947)

(1b) BOWDEN, F.P. and YOFFE, A.D.
Initiation and growth of explosion in liquids and solids.
Cambridge University Press, 1952.

Initiation of explosion when bubbles are present in liquid during impact is due to the adiabatic compression of the bubble and resultant heating.

$$T_2 = T_1 \ (P_2/P_1)\gamma - 1/\gamma$$

where γ = Ratio specific heats

T_1 = Initial temperature

P_1 = Initial pressure

T_2 = Final temperature

P_2 = Final pressure

The final temperature, T_2, depends upon the initial pressure, P_1, and γ, and if a definite high T_2 must be reached, the more difficult the initiation and the higher the final pressure required.

The opposite of the above does not hold true since at lower P_1's, the mass of gas trapped in the bubble becomes less and reduces the quantity of heat produced upon compression.

The presence of gases which normally condense upon compression is not significant since the compression occurs so rapidly that condensation does not occur. There also is evidence that the physico-chemical nature of the gas influences sensitivity.

(2) LIQUID PROPELLANT INFORMATION AGENCY
Liquid propellant test methods: Adiabatic compression sensitivity test.
Test No. 5 (Dec. 1959)

The report describes the initiation of decomposition of liquid monopropellants in the presence of rapidly compressed gas bubbles. The device employs a gas-driven piston which rapidly compresses a gas bubble in contact with a liquid monopropellant. Included is a complete description of the apparatus, a modified version of the test apparatus adopted from the original design submitted to the Committee on Monopropellant Test Methods, "Tentative Specification for the Physical Testing of Monopropellants: The Adiabatic Compression Test", by J.D. Thackrey of the Aerojet-General Corporation.

There are indications that similarity exists between rapid oscillations in pressure with corresponding oscillations in temperature and actual conditions which prevail when a system is exposed to mechanical shock. A definite minimum temperature which can be reached as a result of compressive work may be calculated based on initial temperature and pressure, specific heat ratio and quantity of gas being compressed. The existence of a critical bubble volume is presented.

(3) JOHANSSON, C.H. and SELBERG, H.L.
The ignition mechanism of high explosives.
Appl.Sci.Res. 5A: 439-49 (1956)

Theoretical and experimental results are presented which indicate that an explosive undergoes ignition under compression if the air inclusions contain vapor or particles which upon heating exhibit an exothermic reaction.

Similarly the same "effect is obtained when the explosive forms thin layers between two bubbles or the bubble wall is uneven with projecting convex sections." The paper evaluates the use of a compression cylinder to study the effect of rapid compression which approximates adiabatic compression.

It establishes the importance of the duration of heating with a relationship between time and temperature as a function of initial diameter. The duration of the heating diminishes rapidly with the size of the bubbles and very small bubbles are therefore of no importance as igniting nuclei.

(4) MITCHELL, A.H. and KIRSHNER, H.A.
Compression ignition studies of liquid monopropellants.
AIAA J. 9: 2083-87 (Sept. 1963)

Included is a description of compression-ignition where compression studies were made using a hydraulically driven piston whereby compression ratio, piston travel, and velocity were controlled.

The development of a locked stroke compression drive to minimize the effect of piston bounce is described. Reference is made to a U-tube device and the presence of piston bounce in a U-tube apparatus with the liquid column serving as the piston. This experiment attempted to eliminate the problem of piston bounce in compression ignition noting that the conditions imposed by the tests represent an "extreme test of sensitivity" "since the one way piston does not normally occur in practice."

The experiments included studies in the various attitudes of piston travel: vertical up, vertical down, and horizontal.

(5) MEAD, G.A. (Air Reduction Co., Inc.)
Compression sensitivity of monopropellants.
ARS J. 9: 192-98 (Mar. 1959)

A method of estimating the relative sensitivity of liquid monopropellants by measuring the "severity of conditions necessary to cause initiation" under rapid compression in a piston apparatus and a description of the apparatus is included. The method of analysis elucidates, in terms of pressure rise to cause explosion while relating it to the other physical characteristics of the test, the energy input required to the sample per unit bubble volume.

A comparison of air bubbles and fuel vapor bubbles and their respective sensitivities indicated a lower sensitivity with air. "Critical bubble volume" testing of materials at bubble volumes below critical should be avoided.

(6) GRAY, P. and YOFFE, A.D.
Explosions in liquids initiated from the gas phase: Sensitivity of explosives due to adiabatic heating during sudden compression.
Research (London) 2: 339-40 (1949)

Ignition of explosive vapors that occur in low concentrations under conditions of adiabatic compression are described and give evidence that its behavior confirms the idea of vapor phase inflammation preceding the decomposition in the liquid.

The chemical energy produced by ignition of the explosive vapor augments that energy produced by compression. The importance of the chemical nature of the gas in the bubble is also stressed.

(7) BEANE, G.A.
Evolution of rocket propellant materials: Compatibility testing.
U.S. Air Force Systems Command
Wright Patterson Air Force Base, Ohio
Proj. 3044 (Mar. 1963)
AD 404 491

This paper deals with tests of the compatibility of materials with rocket engine liquid propellants, and a study of a material compatibility with LOX where the possibility of a violent reaction occurs between the two materials upon injection of energy. The choice of tester in this case was a specific impact tester, ABMA type.

2. UNCLASSIFIED LITERATURE SURVEY SUMMARY

The subject matter content of the references listed in the preceding section has provided a background for the study of the fundamental physical and chemical factors involved in the adiabatic testing of high energy explosives. The specific information obtained from these documents include:

1) The study of explosive mechanisms involved in adiabatic compression testing(1,3,6,7).

2) The effect of gas inclusions(1-7).

3) The existence of a critical bubble volume and its relation to sensitivity(1,2,3,5).

4) The effect of the chemical nature of the gas comprising the bubble(1,5,6).

5) The importance of the duration of heating, the relationship between time and temperature(2,3).

6) The effect of piston bounce during adiabatic compression testing(4,2).

7) The "Locked Stroke" tester presented as a severe test of sensitivity(4).

8) Design of equipment used in adiabatic compression tests(2-5).

9) Relationship of pressure rise in test procedure and other physical characteristics involved in adiabatic compression testing(5).

10) Evidence of vapor phase inflammation preceding liquid decomposition(6).

11) The possibility of a violent reaction occurring between two materials upon the injection of energy(7).

3. CLASSIFIED LITERATURE TITLES

The following list of unclassified list of titles was uncovered. Some of the reports were received to assist in the current work. However, since the reports are classified, no information is presented herein.

(1) HUGHES AIRCRAFT CO. (Culver City, Calif.)
Service test evaluation of high energy monofuels. Final Report (U).
Contract No. AF 04(611)-6012
SSD-TR-61-27
AD 329 569 (Confidential)

(2) U.S. BUR. MINES (Pittsburgh, Pa.)
Safety and combustion characteristics of homogeneous and heterogeneous monopropellant systems (U).
AD 331 595 (Confidential)

(3) STAUFFER CHEMICAL CO. (Chauncey, N.Y.)
Gould, J.R.
High energy monopropellants. Summary Report No. 4 (U).
Contract No. NOw 60-0075-c
AD 321 492 (Confidential)

(4) E.I. DUPONT DE NEMOURS (Gibbstown, N.J.)
A mathematical design program for high energy propellant combinations (U).
Contract No. AF 04(611)-7428
AD 326 374 (Confidential)

(5) REACTION MOTORS DIVISION, THIOKOL CHEMICAL CORP. (Denville, N.J.)
High energy storable propellants. Final Summary Report (U).
Contract No. AF 04(611)-5969 (May 26, 1960 - May 25, 1961)
AD 326 085 (Confidential)

(6) U.S. BUR. MINES (Pittsburgh, Pa.)
Mason, C. and Ribovich, J.
Explosion hazards of high energy monopropellant systems (U).
Final Report No. 3839 (June 30, 1961)
AD 327 063 (Confidential)

(7) U.S. BUR. MINES (Pittsburgh, Pa.)
Safety and combustion characteristics of homogeneous and heterogeneous monopropellant systems (U).
Semi-Annual Report No. 3850
AD 329 049 (Confidential)

(8) U.S. BUR. MINES (Pittsburgh, Pa.)
Sensitivity characteristics of high energy monopropellant systems.
Progress Report No. 1
AD 329 048 (Confidential)

(9) U.S. NAVAL ORDNANCE LABORATORY (White Oak, Md.)
Safety information from propellant sensitivity studies (U).
Report No. NOLTR-62-41
AD 329 756L (Confidential)

(10) AIR REDUCTION CO., INC. (Murray Hill, N.J.)
Summary report on test methods for determining operational characteristics of monofuels (U).
Contract No. AF 33(616)-3433 (Feb. 10, 1958)
(Confidential)

(11) BECCO CHEMICAL DIVISION, FOOD MACHINERY and CHEMICAL CORP.
Wesniewski, L.V. and Raleigh, C.W.
Results of card gap and adiabatic compression tests of hydrogen peroxide based low freezing point propellants (U).
Engineering Report LR-E-4
U.S. Navy Contract No. NOAS 53-1034-C (Sept. 1956)
(Confidential)

(12) LIQUID PROPELLANT INFORMATION AGENCY
Fifth monopropellant symposium (Aug. 30 - 31, 1961), Wyandotte, Mich. (U).
Contract No. NOrd 7386
AD 326 905 (Confidential)

(13) NEW YORK UNIVERSITY, COLLEGE OF ENGINEERING
A liquid propellant review. Quarterly Report No. 3 (U).
Contract No. NOw 61-0577-d
AD 326 935 (Confidential)

(14) AIR REDUCTION CO., INC. (Murray Hill, N.J.)
Summary report on advanced test methods for determining operational characteristics of propellants (U).
Contract No. 04(611)-7413 (June 30, 1962)
SSD-TRD-62-215
AD 328 172 (Confidential)

(15) ROCKETDYNE (Canoga Park, Calif.)
Research and development to determine methods to prevent detonation propagation in high-energy monopropellant systems (U).
Final Report No. R-3205
Contract No. AF 04(611)-6066 (Nov. 1961)
SSD-TR-61-35 (Confidential)

(16) COMMERCIAL SOLVENTS CORP. (Terre Haute, Ind.)
Research to evaluate high energy storable liquid rocket fuels. Final Report (U).
Contract No. AF 04(611)-5161 (Feb. 1960 - Jan. 1962)
AD 326 569 (Confidential)

(17) REACTION MOTORS DIVISION, THIOKOL CHEMICAL CORP. (Denville, N.J.)
Quarterly Progress Report
Contract No. NOw 61-0695-c
AD 326 757 (Confidential)

(18) AIR REDUCTION CO., INC. (Murray Hill, N.J.)
Final report on the study of stability of liquid and gaseous ozone-oxygen mixtures (U).
Contract No. AF 33(616)-2709 (Sept. 30, 1957)
(Confidential)

(19) AEROJET-GENERAL CORP (Azusa, Calif.)
Stability of monopropellants (U).
Contract No. AF 33(600)-28890 (Oct. 1955)
(Confidential)

(20) AIR REDUCTION CO., INC. (Murray Hill, N.J.)
Test methods for determining operational characteristics of monofuels (U).
Supplement No. 1
Contract No. AF 33(616)-3433 (Apr. 30, 1958)
(Confidential)

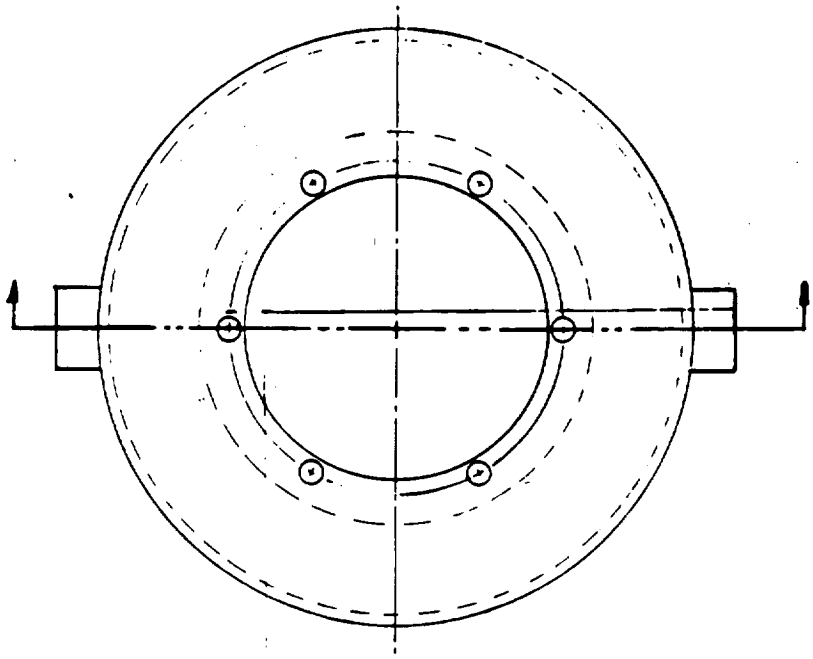
(21) MINNESOTA MINING and MANUFACTURING COMPANY
Evaluation of high energy liquid propellant rocket engine oxidizer and a pilot production of same (U).
Contract No. AF 04(611)-8182 (Feb. 1963)
(Confidential)

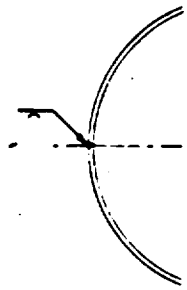
(22) AIR REDUCTION CO., INC. (Murray Hill, N.J.)
Research on high energy rocket oxidizers. Second Technical Progress Report (U).
Contract No. AF 04(611)-9970 (Dec. 15, 1964)
(Confidential)

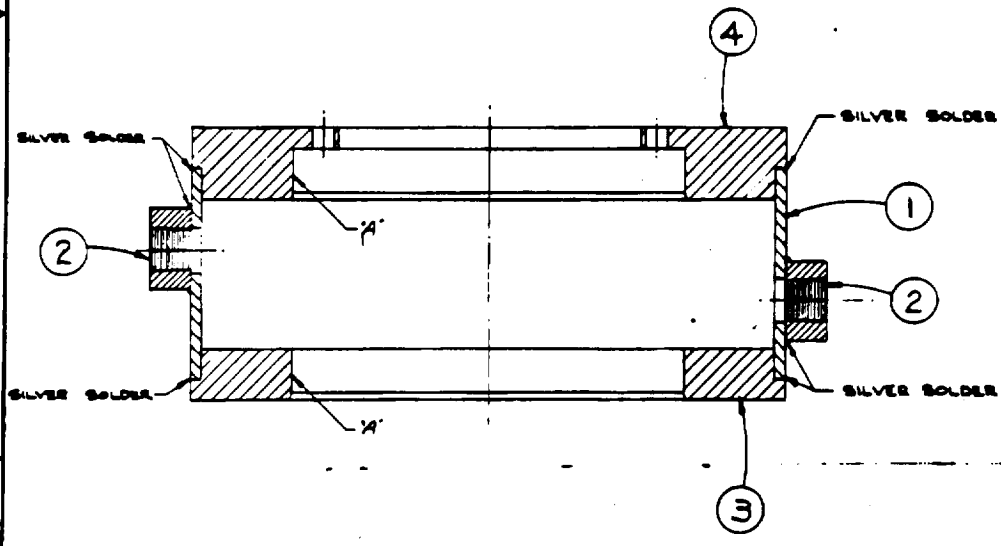
APPENDIX B. Adiabatic Compression Tester Details Drawings

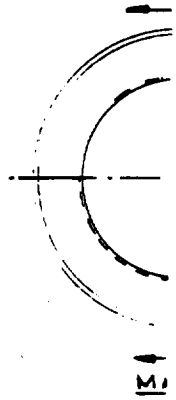
Dwg. No.	
D-898-5460	Outer Coolant Jacket Weldment
C-898-5461	Adiabatic Compression Tester Piston Housing
C-898-5462	Adiabatic Compression Tester Cover
C-898-5463	Adiabatic Compression Tester Test Chamber Housing
C-898-5464	Adiabatic Compression Tester Piston
A-898-5468	Adiabatic Compression Tester Plunger
A-898-5469	Adiabatic Compression Tester Retainer Plate
A-898-5470	Adiabatic Compression Tester Cover Gasket
A-898-5471	Adiabatic Compression Tester Guide Pin
A-898-5472	Adiabatic Compression Tester Spacer
A-898-5473	Adiabatic Compression Tester Rack
A-898-5474	Adiabatic Compression Tester Spring Retaining Nut
A-898-5475	Adiabatic Compression Tester Locking Pin Knob
A-898-5476	Adiabatic Compression Tester Locking Pin
A-898-5521	Adiabatic Compression Tester Diaphragm (Plain)
A-898-5522	Adiabatic Compression Tester Diaphragm
A-898-5754	Adiabatic Compression Tester Insert
A-898-5755	Adiabatic Compression Tester Piston Head
A-898-5756	Adiabatic Compression Tester Piston Sleeve
B-898-5757	Adiabatic Compression Tester Flange
A-898-5758	Adiabatic Compression Tester Sleeve and Insert Assembly
A-898-5942	Adiabatic Compression Tester Flange

Copies of each drawing follow.



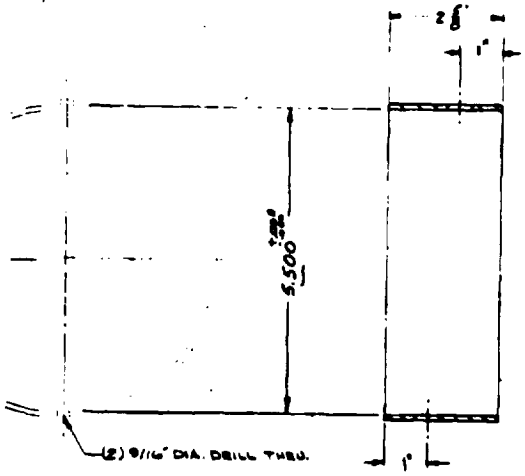




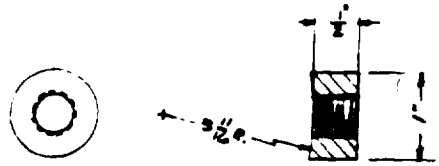


NOTE: 'A' DIM. TO BE CONCENTRIC WITHIN .001 T.I.R.

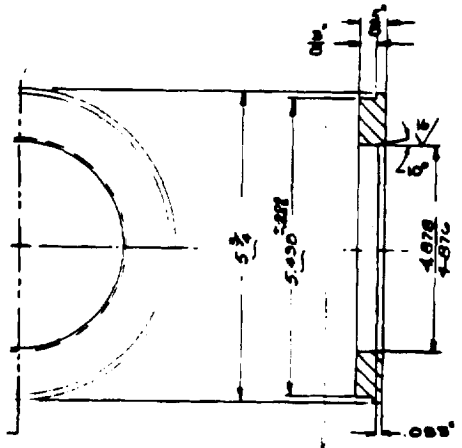
88



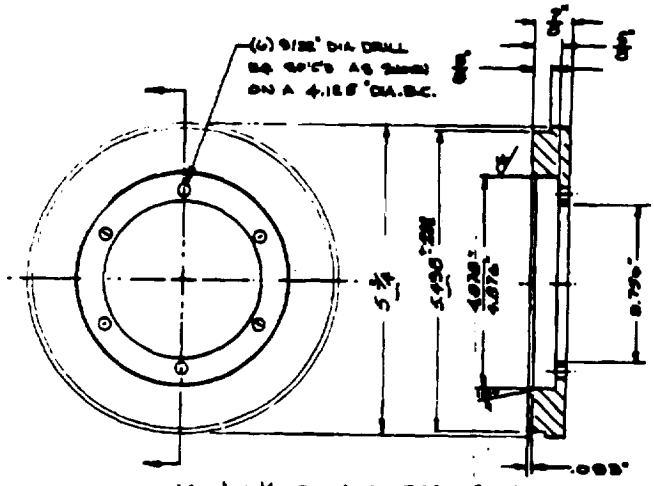
MAT'L: 1/8 THICK, ½ HD NAVAL BRASS (464)
DETAIL OF ITEM NO. 1
SCALE — 1/2" = 1"



MAT'L: MAKE FROM ¼" NPT BRASS HALF COUPLING
DETAIL OF ITEM NO. 2
(2) REQ'D.

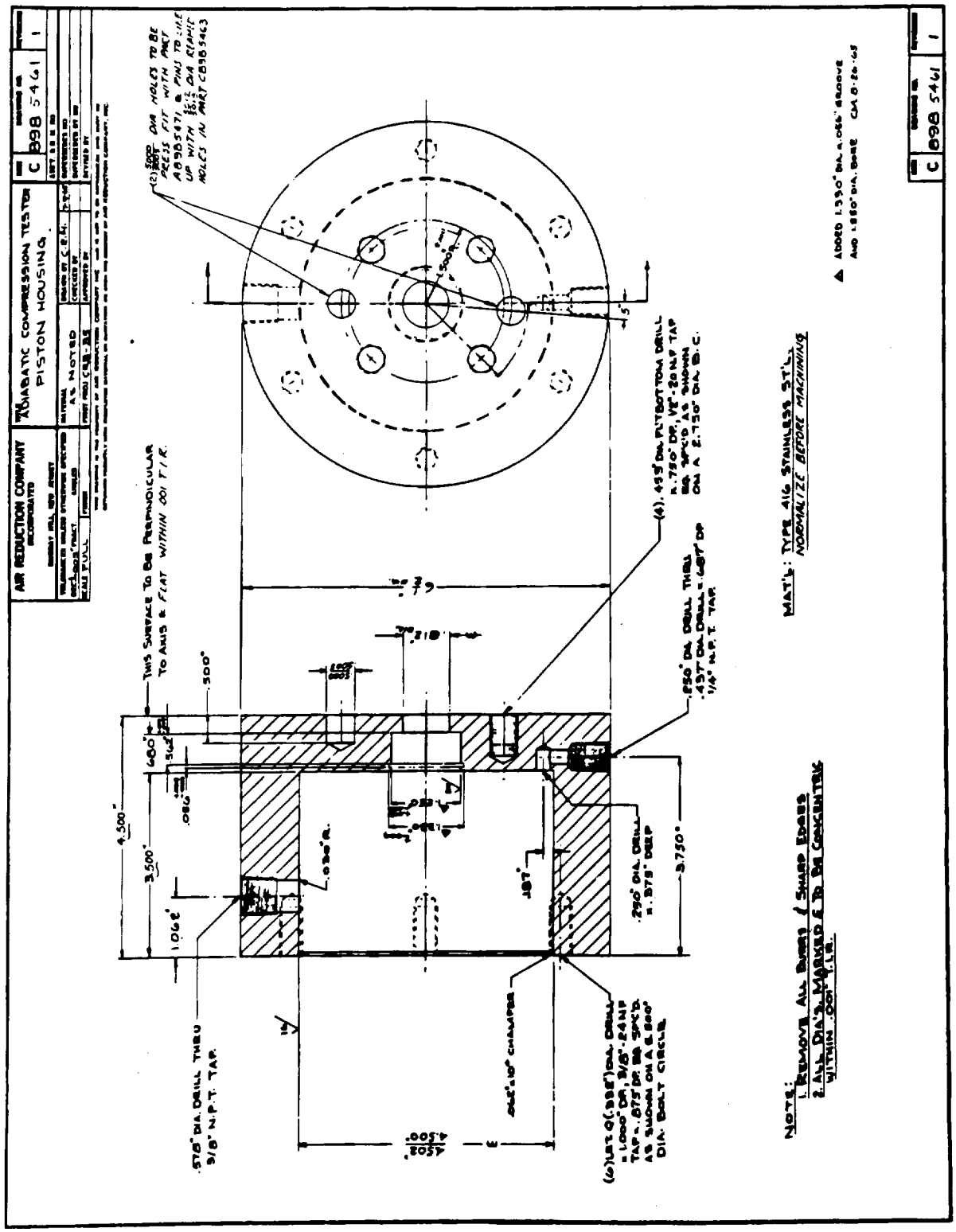


MAT'L: ½ HD NAVAL BRASS (464)
DETAIL OF ITEM NO. 3
SCALE — 1/2" = 1"



MAT'L: ½ HD NAVAL BRASS (464)
DETAIL OF ITEM NO. 4
SCALE — 1/2" = 1"

		TITLE	OUTER COOLANT JACKET WELDMENT
	C.R.M.		
AIRCO	AIR REDUCTION COMPANY, INC.	SIZE D	DRAWING NO. 898 5460



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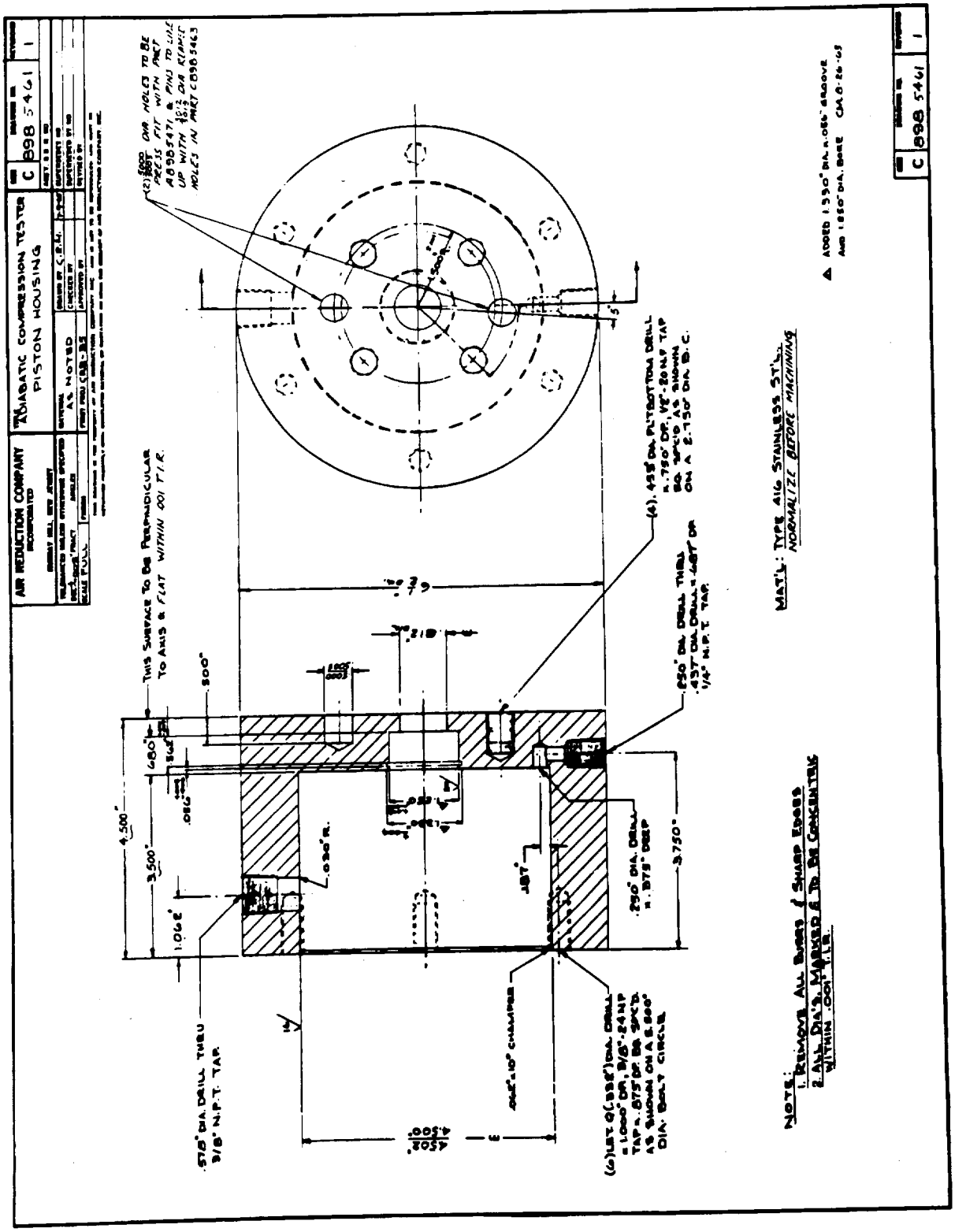

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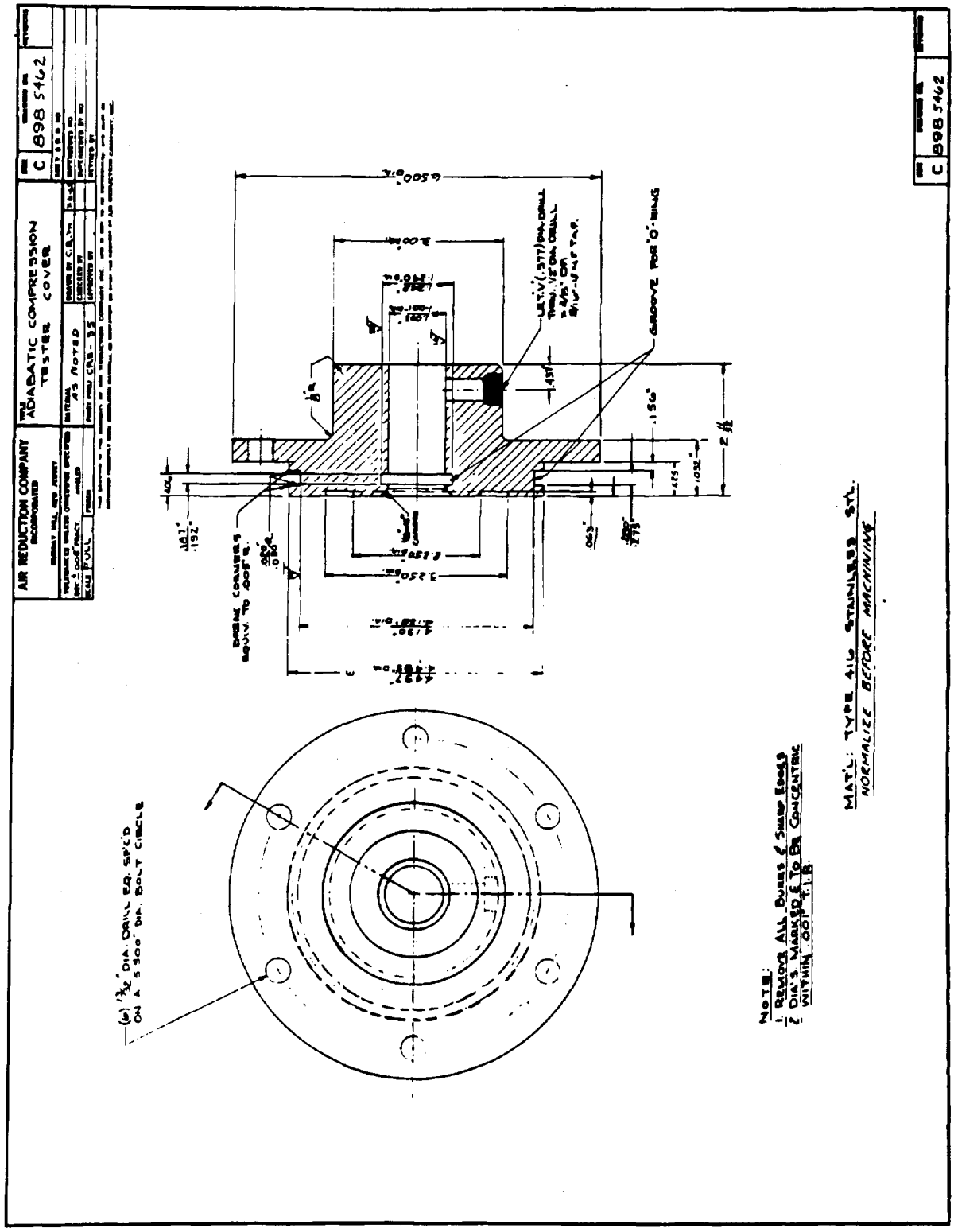

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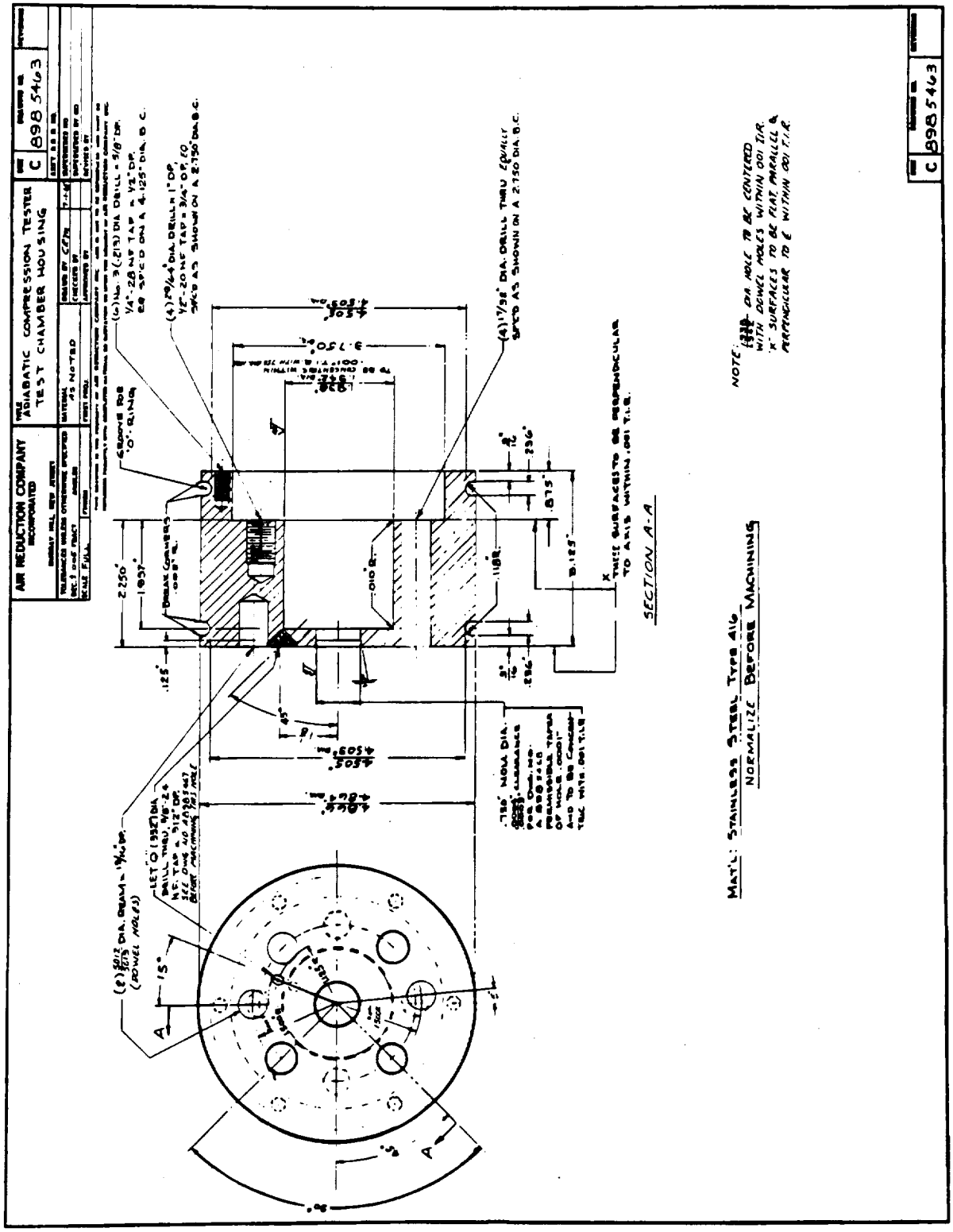

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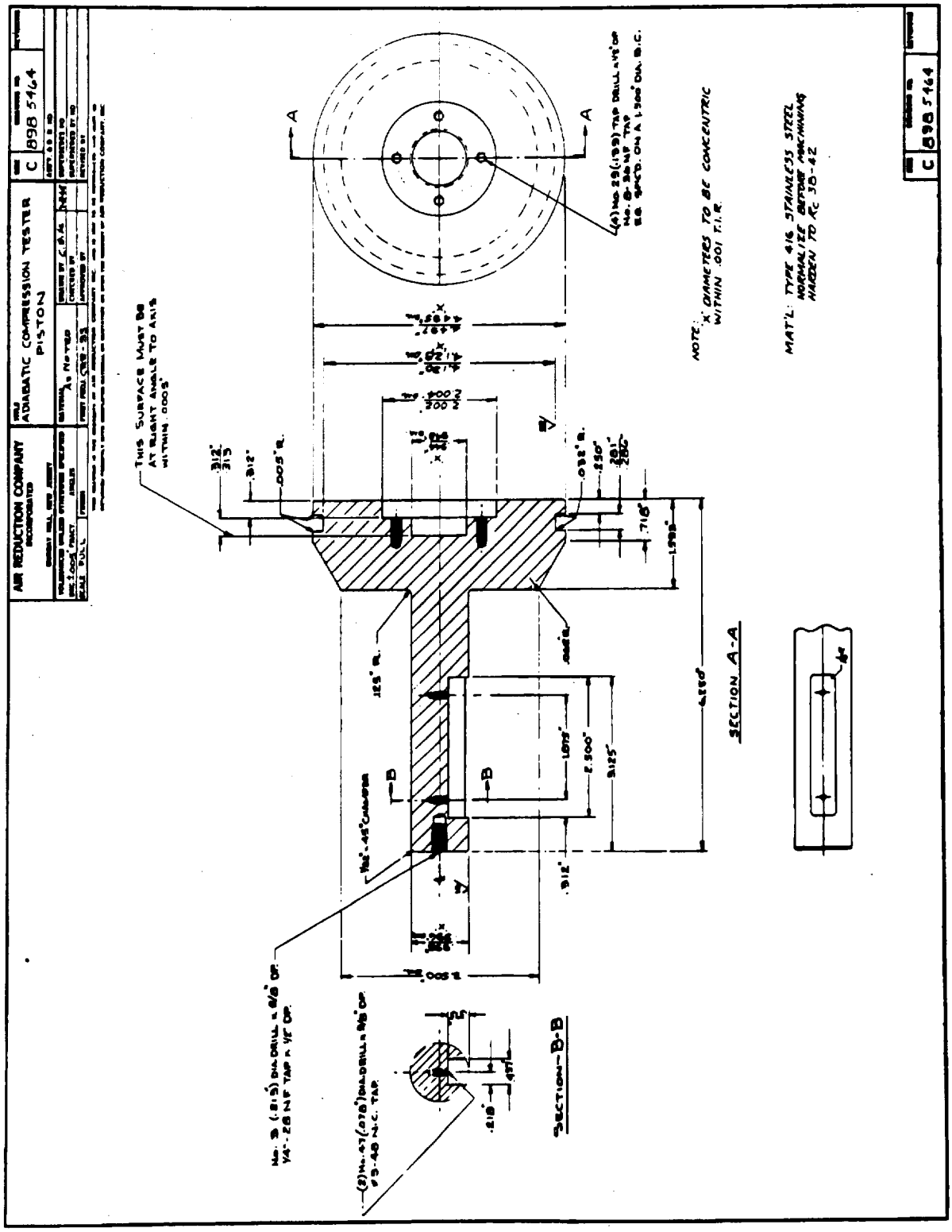

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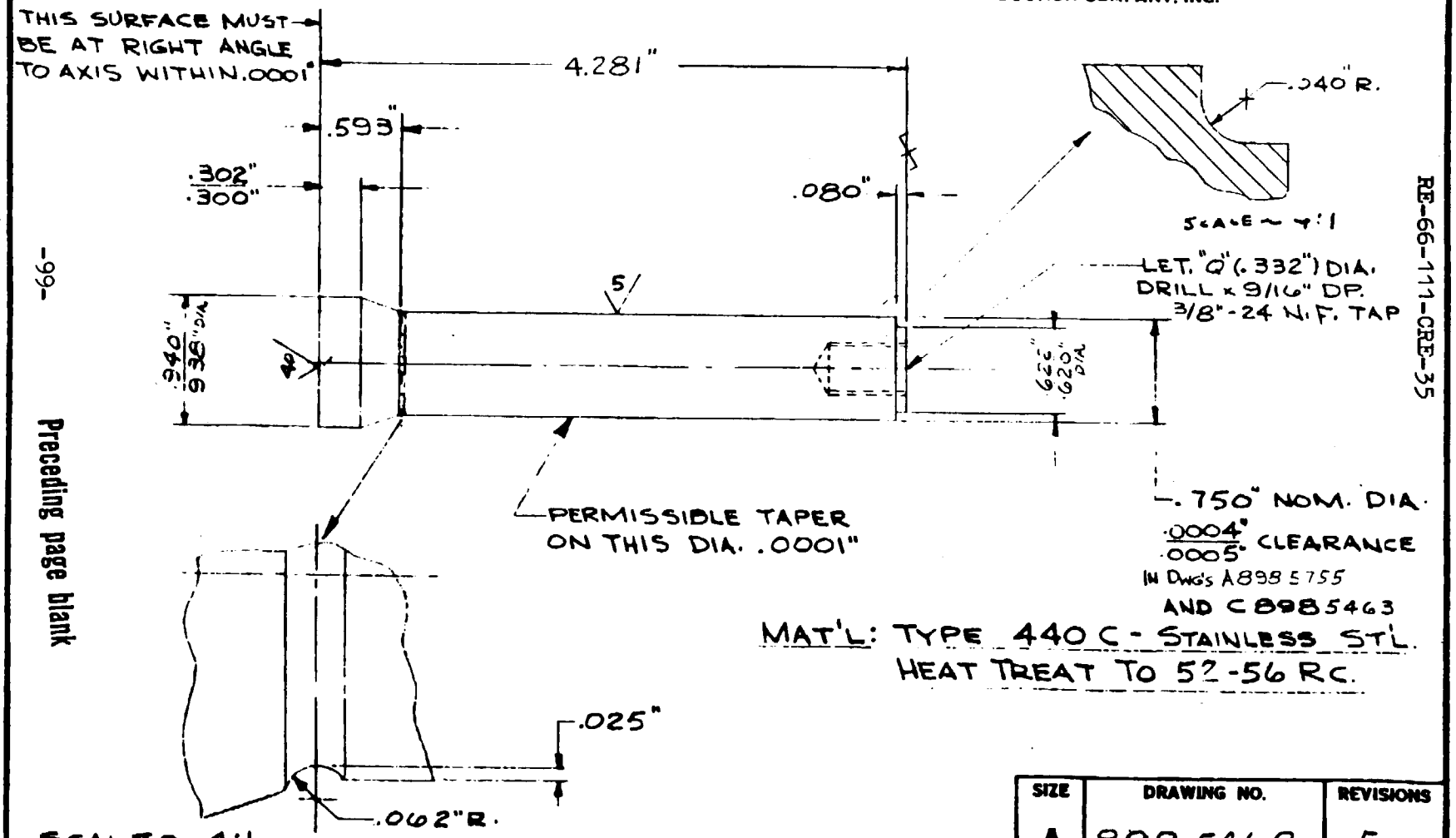
AIR REDUCTION COMPANY INCORPORATED MURRAY HILL, NEW JERSEY	TITLE ADIABATIC COMPRESSION TESTER PLUNGER			SIZE A	DRAWING NO. 898 5468	REVISIONS 5
					ASS'Y. & B. M. NO.	
TOLERANCES UNLESS OTHERWISE SPECIFIED DEC. .005 FRACT. ANGLES	MATERIAL AS NOTED	DRAWN BY C.E.H.	7-9-45		SUPERSEDES NO.	
		CHECKED BY			SUPERSEDED BY NO.	
SCALE FULL FINISH	FIRST PROJ. CCE-35	APPROVED BY			REVISED BY	



RE-66-111-CRE-35

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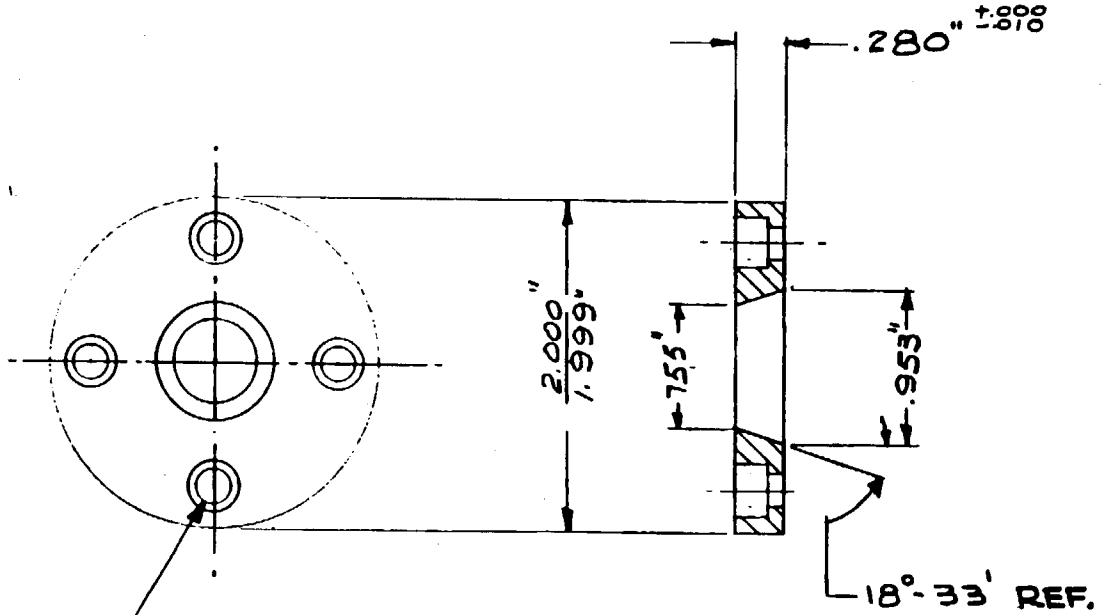
SIZE	DRAWING NO.	REVISIONS
A	898 5468	5

AIR REDUCTION COMPANY INCORPORATED MURRAY HILL, NEW JERSEY	TITLE ADIABATIC COMPRESSION TESTER RETAINER PLATE			SIZE A	DRAWING NO. 898 5469	REVISIONS
				ASS'Y. & B. M. NO.		
TOLERANCES UNLESS OTHERWISE SPECIFIED DEC. ±.005" FRACT. ANGLES	MATERIAL AS NOTED	DRAWN BY CBM	7-9-46	SUPERSEDES NO.		
		CHECKED BY		SUPERSEDED BY NO.		
SCALE FULL	FINISH	FIRST PROJ. CRE-35	APPROVED BY		REVISED BY	



MAT'L: TYPE 416 STAINLESS ST'L.

SIZE	DRAWING NO.	REVISIONS
A	898 5469	

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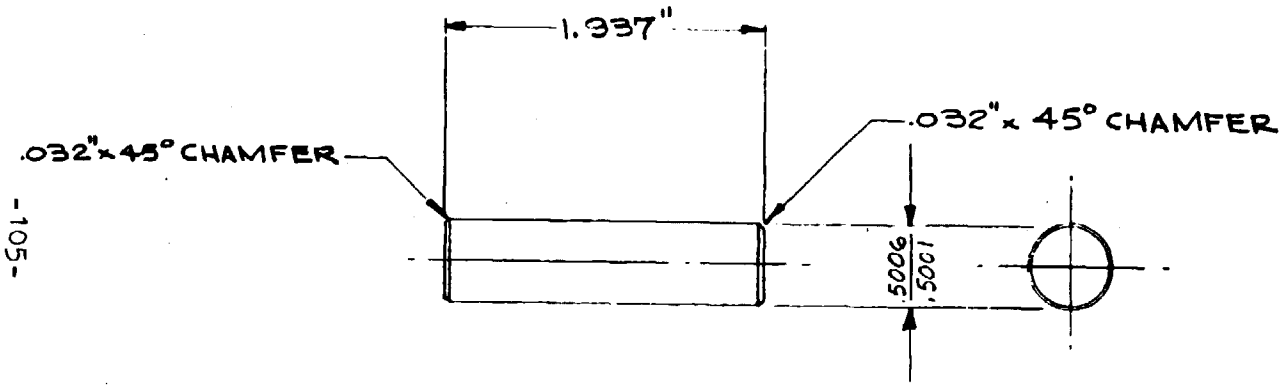
AIR REDUCTION COMPANY INCORPORATED MURRAY HILL, NEW JERSEY	TITLE ADIABATIC COMPRESSION TESTER GUIDE PIN			SIZE A	DRAWING NO. 898 5471	REVISIONS
				ASS'Y. & B. M. NO.		
TOLERANCES UNLESS OTHERWISE SPECIFIED DEC. ±.005" FRACT. ANGLES	MATERIAL AS NOTED	DRAWN BY C.R.M.	7-9-65	SUPERSEDES NO.		
		CHECKED BY		SUPERSEDED BY NO.		
SCALE FULL FINISH	FIRST PROJ. CRE-35	APPROVED BY		REVISED BY		



MAT'L: TYPE 416 STAINLESS ST'L.
(2) REQ'D.

RE-66-111-CRE-35

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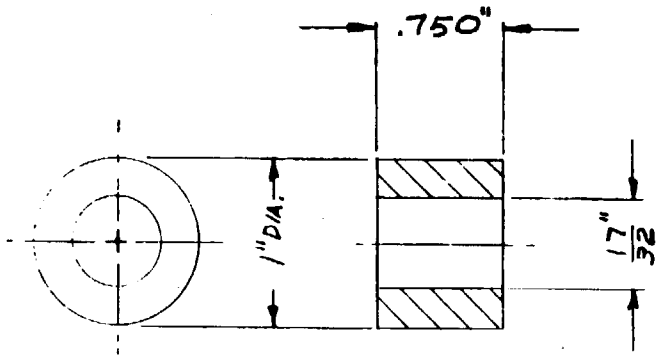
SIZE	DRAWING NO.	REVISIONS
A	8985471	

AIR REDUCTION COMPANY INCORPORATED MURRAY HILL, NEW JERSEY	TITLE ADIABATIC COMPRESSION TESTER SPACER			SIZE A	DRAWING NO. 898 5472	REVISIONS
				ASS'Y. & B. M. NO.		
TOLERANCES UNLESS OTHERWISE SPECIFIED DEC. ±.005" FRACT. ANGLES	MATERIAL AS NOTED	DRAWN BY CEM	7-2-65	SUPERSEDES NO.		
		CHECKED BY		SUPERSEDED BY NO.		
SCALE FULL FINISH	FIRST PROJ. CRE-35	APPROVED BY		REVISED BY		



MAT'L: STAINLESS ST'L TYPE 416

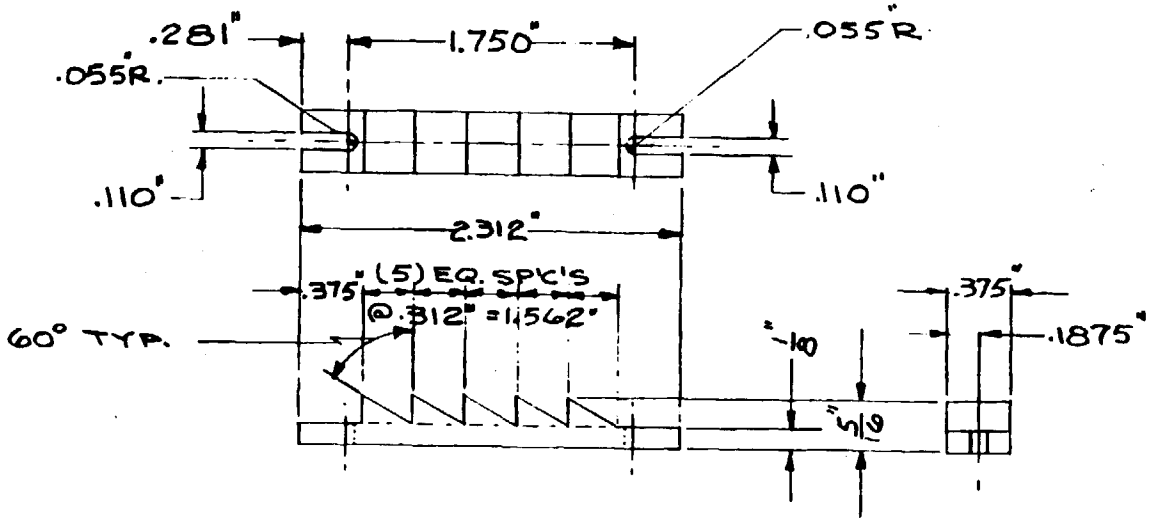
(4) REQ'D

NOTE:

1. FLAT SURFACES TO BE PARALLEL TO ONE ANOTHER WITHIN .0002
2. .750 DIM. ON ALL 4 PIECES TO BE WITHIN .0005 OF ONE ANOTHER

SIZE	DRAWING NO.	REVISIONS
A	8985472	

AIR REDUCTION COMPANY INCORPORATED MURRAY HILL, NEW JERSEY	TITLE ADIABATIC COMPRESSION TESTER RACK			SIZE A	DRAWING NO. 898 5473	REVISIONS
				ASS'Y. & B. M. NO.		
TOLERANCES UNLESS OTHERWISE SPECIFIED DEC. ±.005" FRACT. ANGLES	MATERIAL AS NOTED	DRAWN BY C.E.M.	7-8-65	SUPERSEDES NO.		
SCALE FULL	FINISH	CHECKED BY		SUPERSEDED BY NO.		
	FIRST PROJ.	APPROVED BY		REVISED BY		



MAT'L: LA BELLE SILICON #2 STEEL
AISI-S5 CRUCIBLE STEEL CO.
HEAT TREAT TO R_c 55-60

SIZE A	DRAWING NO. 898 5473	REVISIONS

RE-66-111-CRE-35

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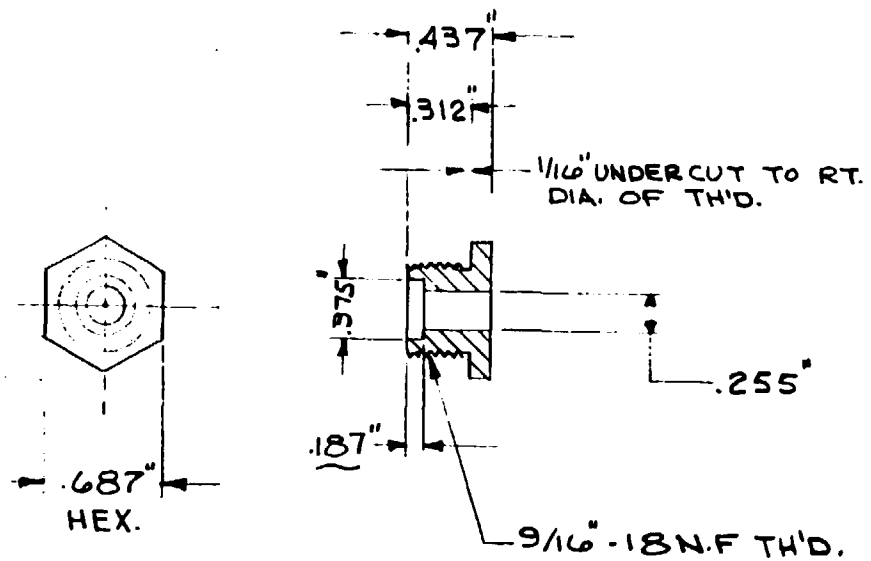
AIR REDUCTION COMPANY INCORPORATED MURRAY HILL, NEW JERSEY	TITLE ADIABATIC COMPRESSION TESTER SPRING RETAINING NUT			SIZE A	DRAWING NO. 898 5474	REVISIONS
					ASS'Y. & B. M. NO.	
TOLERANCES UNLESS OTHERWISE SPECIFIED DEC. ±.005" FRACT. ANGLES	MATERIAL C.R. STEEL	DRAWN BY C.R.M.	7-8-65		SUPERSEDES NO.	
		CHECKED BY			SUPERSEDED BY NO.	
SCALE FULL FINISH	FIRST PROJ. CRE-35	APPROVED BY			REVISED BY	



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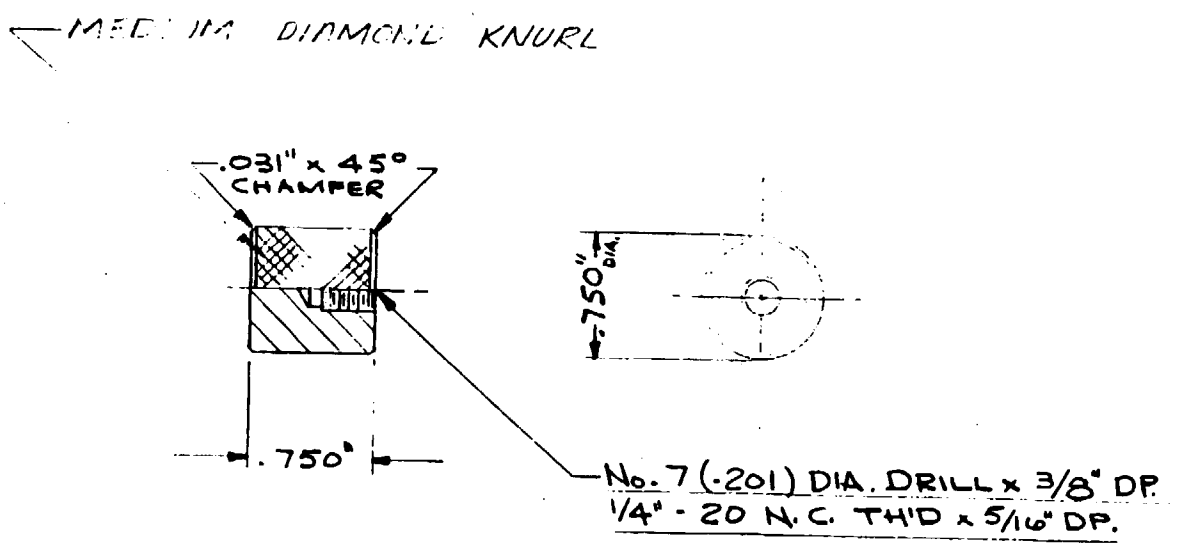
RE-66-111-CRE-35

SIZE	DRAWING NO.	REVISIONS
A	898 5474	

AIR REDUCTION COMPANY INCORPORATED MURRAY HILL, NEW JERSEY	TITLE ADIABATIC COMPRESSION TESTER LOCKING PIN KNOB			SIZE A	DRAWING NO. 898 5475	REVISIONS
TOLERANCES UNLESS OTHERWISE SPECIFIED DEC. ±.005" FRACT. ANGLES	MATERIAL C.R. STEEL	DRAWN BY C.R.M.	7-8-65	ASS'Y. & B. M. NO.		
SCALE FULL FINISH	FIRST PROJ. CRE-35	CHECKED BY		SUPERSEDES NO.		
		APPROVED BY		SUPERSEDED BY NO.		
				REVISED BY		



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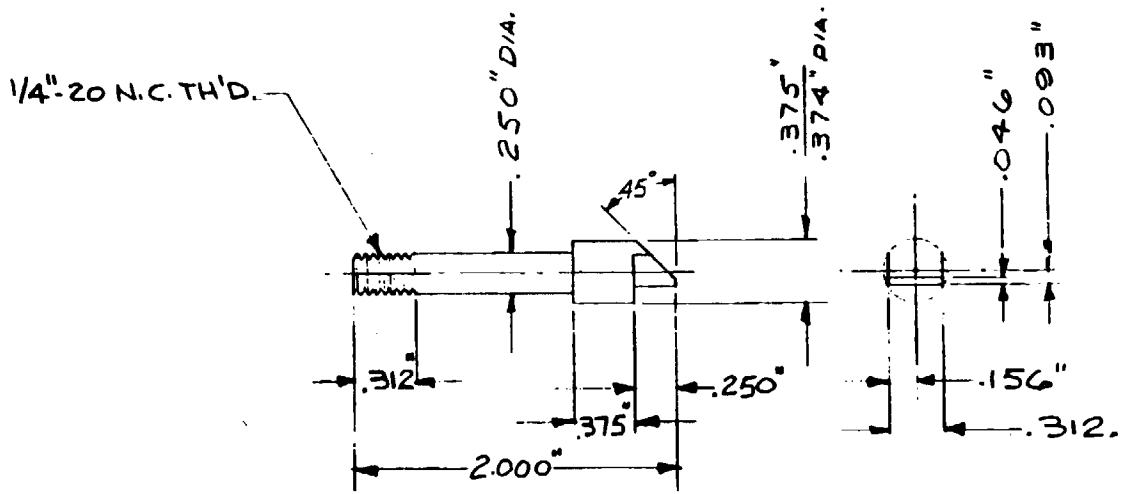
RE-66-111-CRE-35

SIZE	DRAWING NO.	REVISIONS
A	898 5475	

AIR REDUCTION COMPANY INCORPORATED MURRAY HILL, NEW JERSEY	TITLE ADIABATIC COMPRESSION TESTER LOCKING PIN			SIZE A	DRAWING NO. 898 5476	REVISIONS
				ASS'Y. & B. M. NO.		
TOLERANCES UNLESS OTHERWISE SPECIFIED DEC. ±.005" FRACT. ANGLES	MATERIAL AS NOTED	DRAWN BY C.R.M.	7-8-66	SUPERSEDES NO.		
		CHECKED BY		SUPERSEDED BY NO.		
SCALE FULL FINISH	FIRST PROJ. CRE-35	APPROVED BY		REVISED BY		



MAT'L: LA BELLE SILICON #2 STEEL
AISI-55 CRUCIBLE STEEL CO.
HEAT TREAT TO R_c 55-60

RE-66-111-CRE-35

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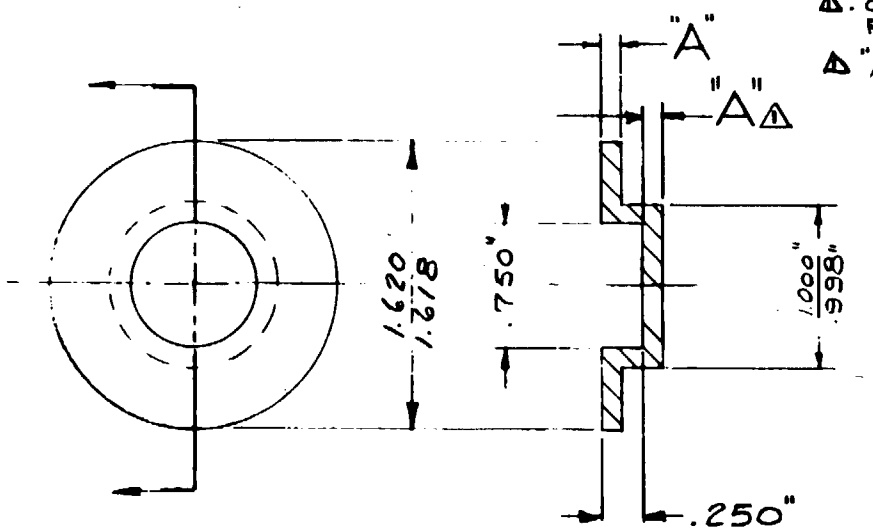
SIZE	DRAWING NO.	REVISIONS
A	898 5476	

AIR REDUCTION COMPANY INCORPORATED MURRAY HILL, NEW JERSEY	TITLE ADIABATIC COMRESSION TESTER DIAPHRAGM (PLAIN)			SIZE A	DRAWING NO. 898 5521	REVISIONS 1
				ASS'Y. & B. M. NO.		
TOLERANCES UNLESS OTHERWISE SPECIFIED DEC. ±.005 FRACT. ANGLES	MATERIAL AS NOTED	DRAWN BY C.R.M.	8-24-66	SUPERSEDES NO.		
		CHECKED BY		SUPERSEDED BY NO.		
SCALE FULL FINISH	FIRST PROJ. CRE-35	APPROVED BY		REVISED BY		

△1 .876" DIA. FOR DIM. A-1 REMOVED
△1 "A" DESIGNATION ADDED C.R.M. 9-23-66



	DIMENSION-"A"		
MAT'L:	-1	-2	-3
TITANIUM ALLOY 5 AL-2 1/2 Sn 6 AL-4 V ALUMINUM ALLOY 5052-H32 MAGNESIUM	0.063"	0.125"	0.250"

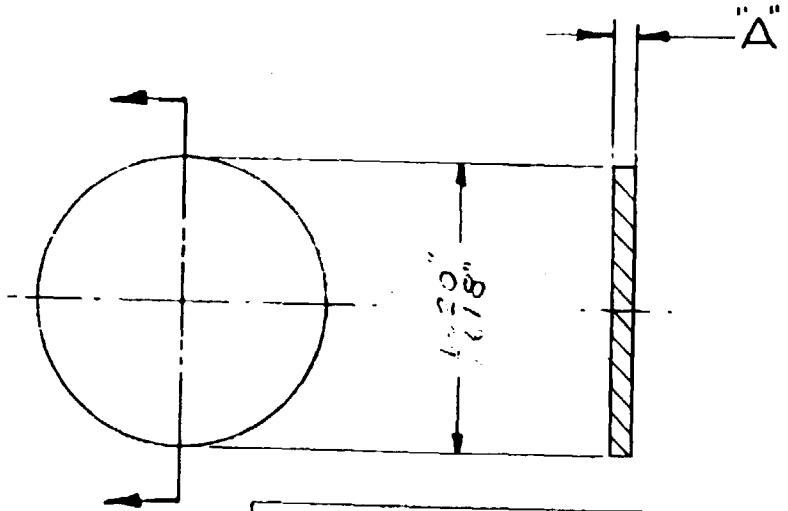
NOTE: THIS PART USED WITH PART No. B 898 5942

SIZE	DRAWING NO.	REVISIONS
A	8985521	1

RE-66-111-CRE-35

-115a-

AIR REDUCTION COMPANY INCORPORATED MURRAY HILL, NEW JERSEY	TITLE ADIABATIC COMPRESSION TESTER DIAPHRAGM			SIZE A	DRAWING NO. 898 5522	REVISIONS
TOLERANCES UNLESS OTHERWISE SPECIFIED DEC. ±.005" FRACT. ANGLES	MATERIAL AS NOTED	DRAWN BY CM	8-24-66	ASS'Y. & B. M. NO.		
SCALE FULL FINISH		CHECKED BY		SUPERSEDES NO.		
	FIRST PROJ. CRE-35	APPROVED BY		SUPERSEDED BY NO.		
				REVISED BY		

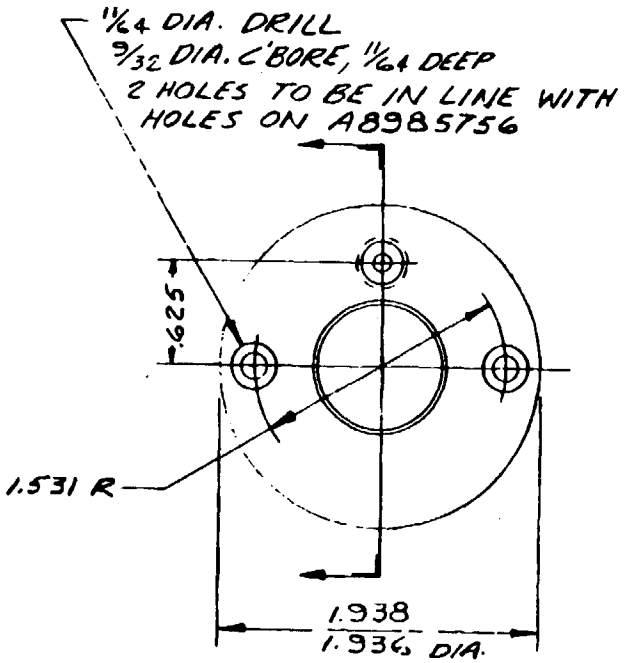


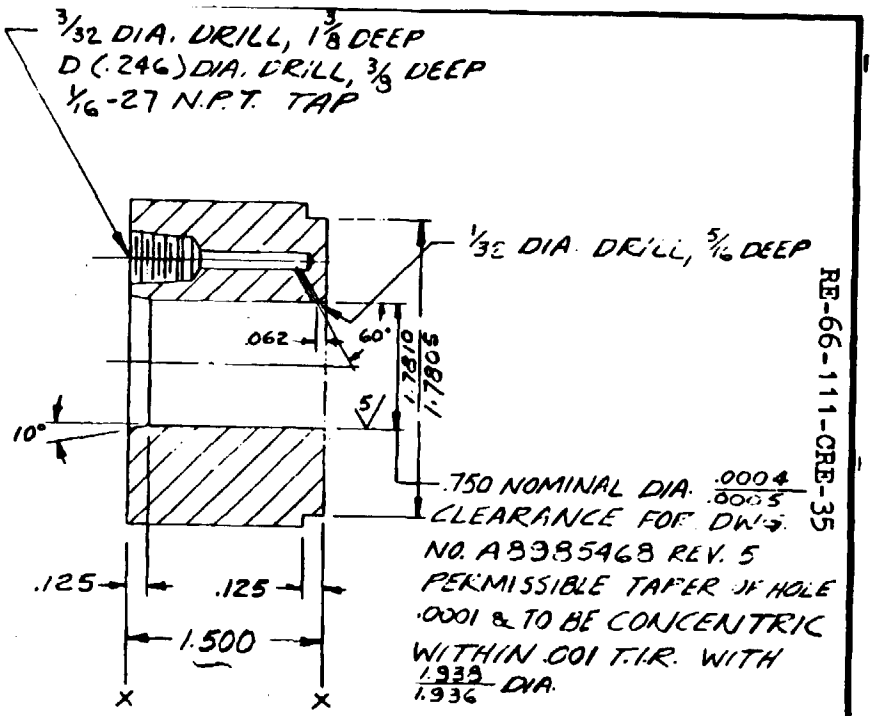
MAT'L:	DIMENSION - "A"						
	-1	-2	-3	-4	-5	-6	-7
TITANIUM ALLOY 5AL-2½ Sn 6AL-4V ALUMINUM ALLOY 5052-H32 MAGNESIUM	0.005"	0.010"	0.025"	0.032"	0.063"	0.125"	0.250"

SIZE	DRAWING NO.	REVISIONS
A	898 5522	

-115b-

RE-66-111-CRE-35





RE-66-111-CRE-35

NOTE:
SEE DWG. A8985758 FOR FINISHING .750 I.D. & 1.938/1.936 O.D.

MAT.
#440 STAINLESS STEEL

SYM. REV.	DATE	REVISIONS	DWN.	CHK.	APP.	APP.
ORIG.	5/16/66	DESIGNED BY PB				

TOLERANCES
DEC. ±.005
FRACT. ±1/64
ANGLES

TITLE ADIABATIC COMPRESSION TESTER INSERT

SCALE 1:2

SUPERSEDES NO.

SUPERSEDED BY NO.

B. M. NO.

DWG. LIST

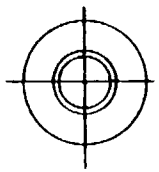
SIZE A

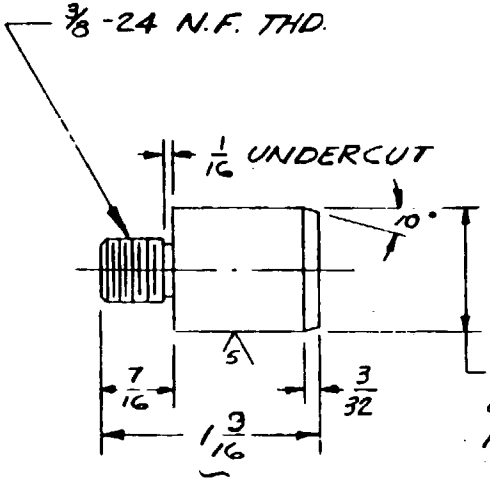
DRAWING NO. 8985754

REV.

SHEET NO. OF SHEETS

AIRCO AIR REDUCTION COMPANY, INC.
MURRAY HILL, NEW JERSEY





.750 NOM. DIA. .0004/.0005 CLEARANCE IN DWG'S. A8985754 & A8985756 SEE NOTE

NOTE:
PIECE IS TO BE ASSEMBLED WITH A8985468 REV.5 & .750 DIA. WILL BE FINISHED IN PLACE IN ORDER TO HAVE PERFECT ALIGNMENT

MAT.
#440C STAINLESS STEEL
HEAT TREAT TO 52-56 RC

RE-66-111-CRE-35

SYM. REV.	DATE	REVISIONS	DWN.	CHK.	APP.	APP.
ORIG.	3/14/66	DESIGNED BY PB				

TOLERANCES
DEC.
FRACT.
ANGLES

TITLE ADIABATIC COMPRESSION TESTER PISTON HEAD

SCALE 1:1

SUPERSEDES NO.

SUPERSEDED BY NO.

B. M. NO.

DWG. LIST

AIRCO

AIR REDUCTION COMPANY, INC.

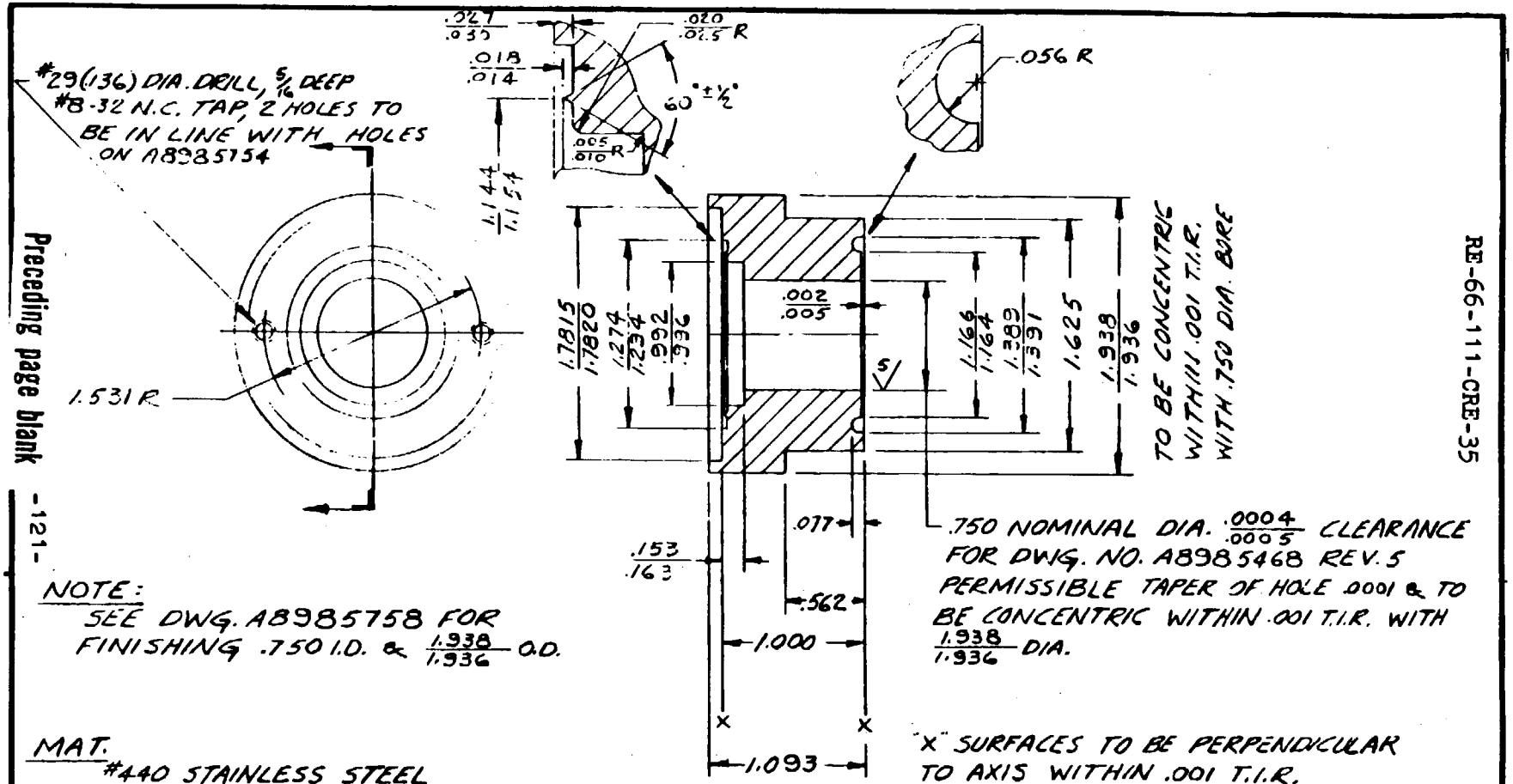
MURRAY HILL, NEW JERSEY

SIZE A

DRAWING NO. 8985755

SHEET NO. OF SHEETS

REV.



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RE-66-111-CRE-35

TO BE CONCENTRIC WITHIN .001 T.I.R. WITH .750 DIA. BORE

.750 NOMINAL DIA. $\frac{.0004}{.0005}$ CLEARANCE FOR DWG. NO. A8985468 REV. 5 PERMISSIBLE TAPER OF HOLE .0001 & TO BE CONCENTRIC WITHIN .001 T.I.R. WITH $\frac{1.938}{1.936}$ DIA.

NOTE:
SEE DWG. A8985758 FOR FINISHING .750 I.D. & $\frac{1.938}{1.936}$ O.D.

MAT.
#440 STAINLESS STEEL
HEAT TREAT TO 52-56 RC

"X" SURFACES TO BE PERPENDICULAR TO AXIS WITHIN .001 T.I.R.

SYM. REV.	DATE	REVISIONS	DWN.	CHK.	APP.	APP.
ORIG.	3/11/64	DESIGNED BY PB				

TOLERANCES
DEC. ±.005
FRACT. ± 1/64
ANGLES

TITLE ADIABATIC COMPRESSION TESTER
PISTON SLEEVE

SCALE 1:1

SUPERSEDES NO.

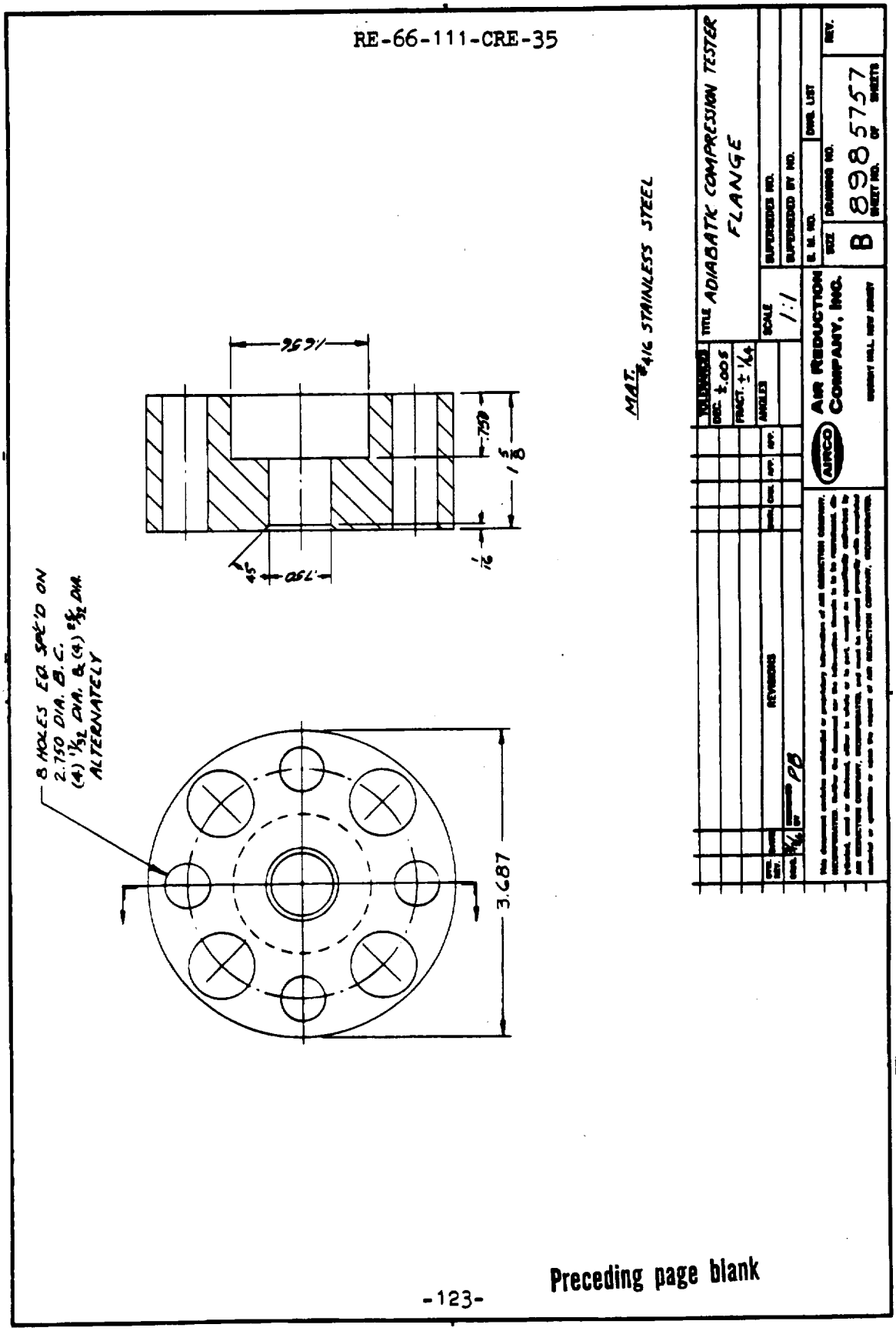
SUPERSEDED BY NO.

B. M. NO. | DWG. LIST

SIZE A | DRAWING NO. 8985756 | REV.

SHEET NO. OF SHEETS

AIRCO
AIR REDUCTION COMPANY, INC.
MURRAY HILL, NEW JERSEY



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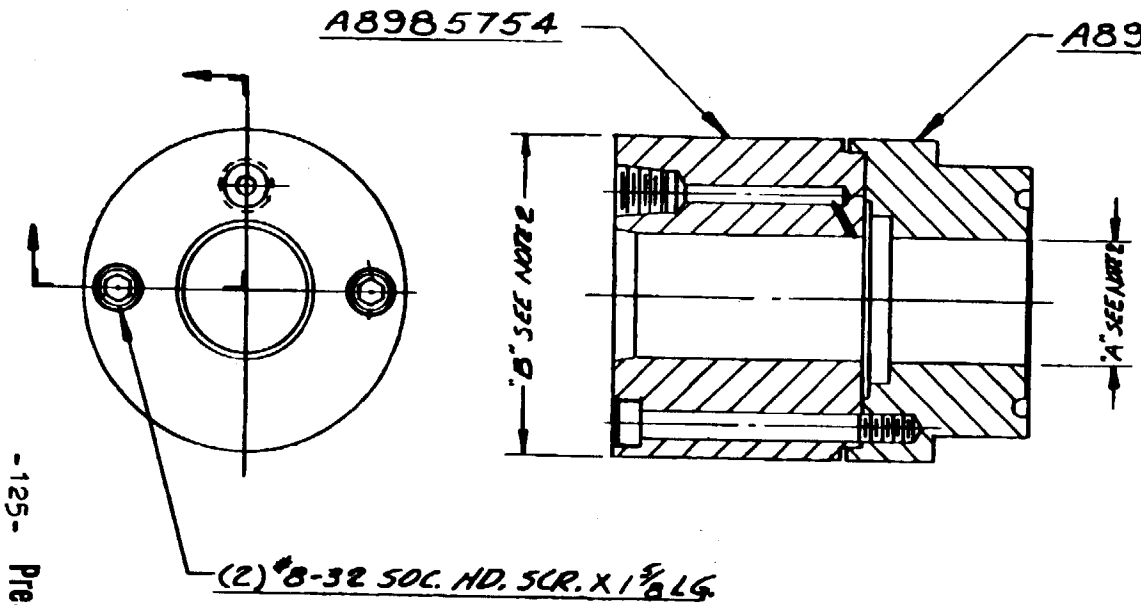

RE-66-111-CRE-35

NOTE:
1. SCREW THESE TWO PIECES TOGETHER AND THEN FINISH "A" I.D. & "B" O.D.
2. SEE DWG. A8985754 & A8985756 FOR INSTRUCTIONS IN FINISHING "A" & "B"

SYM. REV.	DATE	REVISIONS	DWN.	CHK.	APP.	APP.
ORIG.	7/64	DESIGNED BY PB				

TOLERANCES
DEC.
FRACT.
ANGLES

TITLE ADIABATIC COMPRESSION TESTER SLEEVE & INSERT ASS'Y

SCALE 1:1

SUPERSEDES NO.

SUPERSEDED BY NO.

E. M. NO. | DWG. LIST

SIZE A | DRAWING NO. 8985758 | REV.

SHEET NO. OF SHEETS

AIRCO
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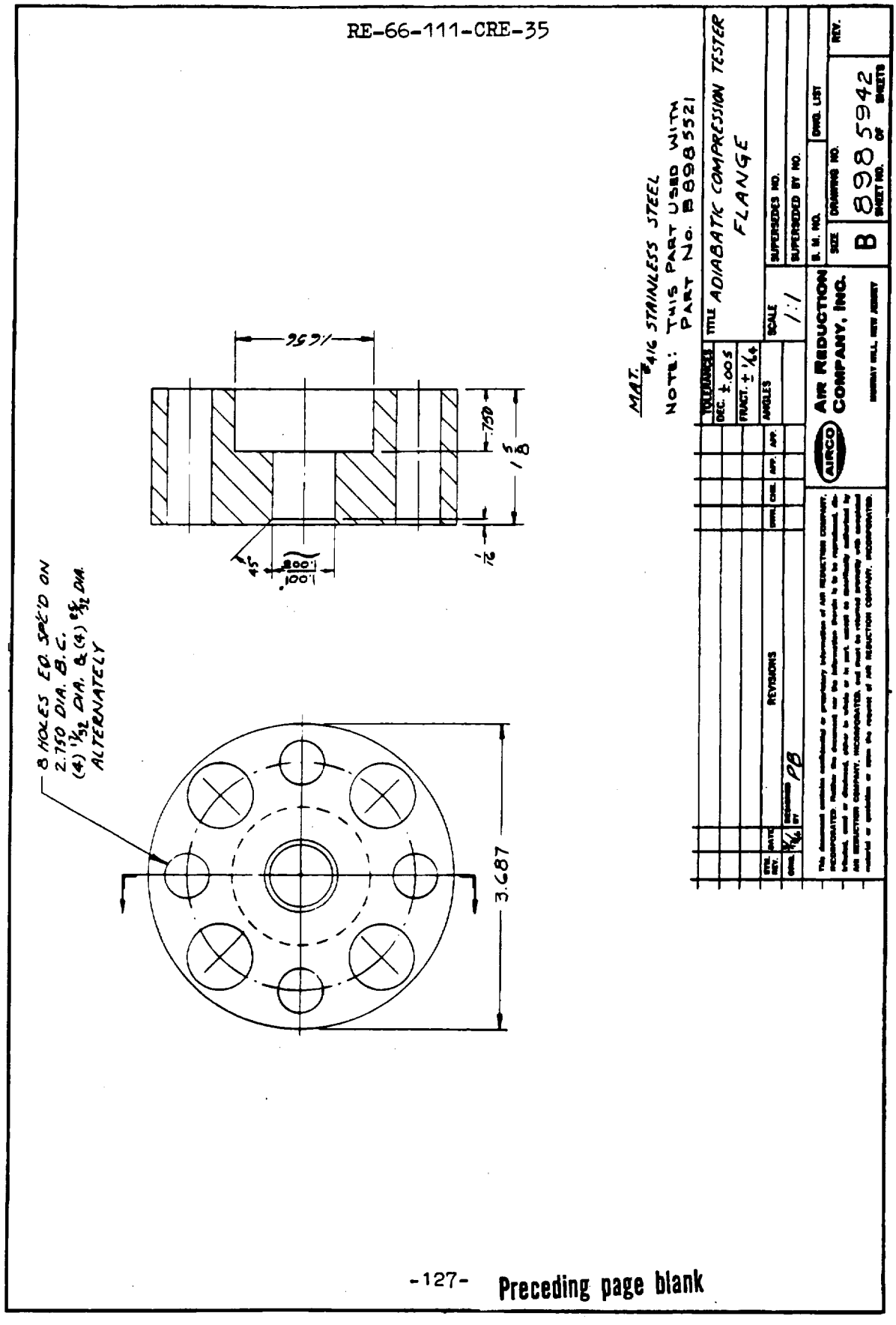

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1. AN EXAMPLE PROCEDURE FOR LOADING LIQUID OXYGEN INTO THE TEST CHAMBER

First determine the initial volume of the test cavity by physically measuring the test cavity (piston retracted to expose the filling port). Measure the depth of the chamber from the face of the retracted piston to the bottom face of the diaphragm. Also measure the diameter of the cavity. Calculate the volume of the test cavity as follows:

Chamber diameter = 0.750 in.

Chamber depth = 1.0 in.

Therefore, the chamber volume = $\frac{\pi d^2}{4}(h) = \frac{3.14(0.750)^2}{4}(1.0) =$

$= 0.442$ cu.in.

and converting 1 cu.in. = 16.39 cc.

0.442 cu.in. × 16.39 cc./cu.in. = 7.2 cc.

Another method for determining the initial test cavity volume is to fill the chamber with a measured quantity of water without the diaphragm in place. Fill the cavity using a pipette and record the amount of water used.

Once you have established the test cavity volume in the chamber loading position, you now select what volume of liquid oxygen you want to test and with what bubble volume. As an example:

Fill the test cavity with 1 g. of liquid oxygen and, leaving a bubble volume of 1 cc. in the cavity,

1) Determine the total volume for the sample. The density of liquid oxygen at 1 atm. pressure = 1.14 g./cc. The volume occupied by 1 g. of liquid oxygen = 1 g. ÷ 1.14 g./cc. = 0.876 cc. The bubble volume is 1 cc.; therefore, the total volume will be 1 cc. + 0.876 cc. = 1.876 cc.

2) Determine piston position to give the calculated total volume. The piston displacement necessary to obtain the calculated total volume is the fraction of the total volume times the initial chamber depth.

$$\frac{\text{cc. at desired volume}}{\text{cc. at charging volume}} \times \text{Chamber depth at charging volume} = \text{Depth at desired volume}$$

$$\frac{1.876 \text{ cc.}}{7.2 \text{ cc.}} \times 1.0 \text{ in.} = 0.256 \text{ in.}$$

The piston, therefore, will be moved forward
1.0 in. - 0.256 in. = 0.744 in.
from the initial loading position after charging with the oxygen. This piston positioning is referenced to the piston displacement calibration on the oscillograph.

3) Charging the desired quantity of oxygen into the chamber. In order to charge the desired quantity of oxygen into the test chamber, it is necessary to specify the pressure, temperature, and volume at the charging condition (ambient temperature). Note the typical sample:

Ambient temperature = 70°F (21°C)

Total chamber volume = 7.2 cc.

Quantity of liquid oxygen to be charged = 1 g.

$$\text{Specific volume} = \frac{\text{Total chamber volume available}}{\text{Quantity to be charged}} =$$

$$\frac{7.2 \text{ cc.}}{1 \text{ g.}} = 7.2 \text{ cc./g.}$$

From the thermodynamic properties of oxygen (temperature entropy) at 21°C and a specific volume of 7.2 cc./g., the pressure is 100 atm. or 1470 p.s.i.a. In this example, the chamber cavity of 7.2 cc. volume at ambient conditions (21°C) must be pressurized to 1470 p.s.i.a. to obtain 1 g. of gaseous oxygen charge in the chamber. Upon condensing the gaseous oxygen to -297°F and 1 atm., the resultant liquid oxygen volume will be 0.866 cc. The bubble volume is the difference between the final volume and the liquid volume (1.876 cc. - 0.987 cc. = 1 cc.). The oxygen content of the bubble at cooldown to -297°F is determined by knowing the specific volume at 1 atm. and -297°F. Specific volume determined from thermodynamic oxygen charts at -297°F and 1 atm. equals 225 cc./g.; and, since the desired bubble volume is 1 cc., the oxygen content in grams is

$$\frac{1.0 \text{ cc.}}{225 \text{ cc./g.}} = 0.0044 \text{ g.}$$

If desired, the residual quantity may be used to correct the liquid volume of oxygen. Density of liquid oxygen = 1.14 g./cc.; therefore,

$$0.0044 \text{ g.} \div 1.14 \text{ g./cc.} = 0.0038 \text{ cc.}$$

1136

2. EFFECT OF RESIDUAL VOLUME RESULTING IN THE MECHANICAL CLEARANCE BETWEEN THE END OF THE PISTON AND DIAPHRAGM IN THE TEST CAVITY

The clearance between the piston and diaphragm is maintained to prevent contact between the piston and diaphragm when the rig is fired. This clearance is a function of the minimum quantity of oxygen desired to be charged into the cavity and the maximum pressure desired upon compression.

EXAMPLE

If the minimum quantity of oxygen desired to be charged at 1 atm. is 7.2 cc. and the final pressure desired is 15,000 p.s.i.a. $\cong$ (1100 atm.), the compression ratio will equal 1100/liter. The charge in volume is, therefore, approximately 1100. We see that 1/1100 of 7.2 cc. = 0.0065 cc. which is the maximum clearance volume in order to achieve 15,000 p.s.i.a. To determine this clearance in inches from the piston face to the diaphragm face, we see

$$0.0065 \text{ cc.} \times 1/16.39 \text{ cc./cu.in.} = 0.0004 \text{ cu.in.}$$

and

$$0.0004 \text{ cu.in.} = \frac{\pi d^2}{4}(h) = \frac{3.14(0.75)^2}{4}(h)$$

The clearance h in inches $\cong$ 0.001. Since this clearance is difficult to gauge, we recommend a clearance of 0.030 in. This clearance will result in calculated minimum gas quantity at room temperature of 0.216 cc. This is obtained from the clearance volume

$$\frac{\pi d^2}{4}(h) = (0.442 \text{ sq.in.})(0.03 \text{ in.}) =$$

$$0.01326 \text{ cu.in.} \times 16.37 \text{ cc./cu.in.} = 0.216 \text{ cc.}$$

Therefore, with an initial volume of 7.2 cc., the volume change represents a compression ratio of

$$\frac{7.2 \text{ cc.}}{0.216 \text{ cc.}} = 33.4:1$$

Therefore, if the maximum pressure desired is 15,000 p.s.i.a., the initial pressure will be 450 p.s.i.a. This clearance residual only presents a problem in the cases where:

1) Only small gas volumes are compressed, and

2) Where liquid volume is less than the clearance volume.

R. Newcombe
Project Engineer

Approved:

W.B. Moen
Assistant Chief Engineer

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RE-66-111-CRE-35
Suppl. 1

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